

A convenient, reliable and costeffective solution for GPC-SEC analysis

Technical Note

Introduction

Gel Permeation Chromatography (GPC), also referred to as Size Exclusion Chromatography (SEC) is an important tool to characterize polymers. GPC-SEC can be applied to oligomers with a molecular weight of less than 2000D but also to polymers with molecular weights of several millions. The main results are molecular weight averages and molecular weight distribution curves which characterize a polymer's properties.

The GPC data analysis software for the Agilent ChemStation is part of the Agilent 1100 Series GPC system which provides a complete solution for convenient, cost-effective and precise routine GPC-SEC analysis.

The GPC-SEC software is an add-on package for the proven Agilent ChemStation for LC systems and designed to operate with concentration-sensitive detectors such as refractive index and UV-visible detectors. Signals of other detectors, for example, evaporative light scattering detector, can be processed using the Agilent 35900E analog to digital converters. Special detectors that provide absolute molecular weights, for example, viscosimetric or laser light scattering detectors need to be operated with the software that comes with these detectors.



General Description

The Agilent 1100 Series and HP 1050 Series instrument control, data acquisition and conventional peak analysis (for example, percent area reports) is performed by the standard HPLC ChemStation, whereas dedicated GPC-SEC evaluation and reporting is done by the GPC data analysis software. The chromatographic raw data can be evaluated interactively or unattended and automatically. The software simplifies retrieval of GPC data and reconstitutes results, including average molecular weights, M_n, M_w, M_z, M_p, M_v, polydispersity D, differential and integral molecular weight distribution. The software allows internal standard and detector delay corrections for multi-detector configurations. A separate software window provides access to the tasks in narrow standard and universal calibration. Wizards guide you through universal and integral calibration. The quality of the calibration curve can be checked by reviewing the percentage deviation for each calibration point individually or by quickly checking the polynomial coefficients and the least square fit chi square.

The software comes with validation and automated instrument verification procedures, and is fully compliant with ISO/EN 13885, DIN55672 and ASTM D 5296-97 GPC standard procedures. All features of the GPC data analysis software are set up in logical units which prevents the user from 'getting lost between the many windows and options'. This is achieved by displaying the following items simultaneously on a single screen

- raw chromatogram with sample name and the detector in use,
- flow-rate-corrected chromato gram, called elugram,
- molar mass distribution, and molecular weight data.

Consequently, the user will notice and overcome evaluation mistakes quickly and easily. With a single keystroke switching between different views or returning to a standard screen configuration is possible.

The Agilent GPC data analysis software offers three different modes of GPC data processing:

- manual reprocessing of individual data files from the standard ChemStation data analysis screen,
- interactive reprocessing of GPC runs for fine-tuning demanding samples in the GPC data analysis screen, and
- automatic GPC data processing for single runs and sequences (on-line and off-line) from the ChemStation run control screen.

For data review the samples can be searched via a selection of criteria, for example, date, operator, solvent. The software is capable of multitasking. While one sequence of samples is analyzed and new data are acquired and reported, stored data files can be loaded and reprocessed starting from the standard Agilent ChemStation data analysis or from the interactive GPC data analysis screen using different calibration files.

Key Software Features

Method setup

The GPC data analysis software is seamlessly integrated into the Agilent HPLC ChemStation. Therefore, the software is easy to learn, especially for users already familiar with the Agilent ChemStation. Figure 1 shows how it directly uses the ChemStation for Agilent 1100 Series instrumentation control and its data acquisition engine.

The chromatographic raw data are automatically transferred to the GPC data analysis software runby-run for state-of-the art GPC data evaluation. Therefore, in addition to the GPC report a normal HPLC report can be obtained simultaneously for each analysis. This is an advantage when both polymer characterization and quantitative determination of monomers and oligomers is required.



Figure 1 Seamless integration of GPC data analysis software into the Agilent ChemStation

The software retrieves all necessary analysis parameters, for example, the flow rate from the Agilent 1100 Series or 1050 Series pumps. Flow rates from third party or Agilent 1090 Series solvent delivery systems can be entered manually to calculate elution volumes. The operator controls the data evaluation primarily via the *GPC Settings* window (figure 2). Here the user specifies

GPC Settin Calibration Report Settings Interactive screen reviev File : demo_gpc.CA Print results Reference Detector for Recalibration Configure Print BID1 A. Befractive Index Signal Reference Detector for Internal Standard Co C Print Preview Default
 Printer A -🔽 Text File BUse Internal Standard Corr Max Deviation A Slice.txt Reference Position : % min Report Subsets as Mass Fraction at M [D] Molar Mass at I(M) [%] Ø Detector Configuration Delay [min] Color DAD1 A, Sig=254,4 Ref=550,100 Θ + + + DAD1 B, Sig=230,4 Ref=550,100 RID1 A. Refractive Index Signal none 0 More GPC Settings Cancel OK



- 1. Selected calibration file
- 2 Automatic calibration
- 3. User-selectable internal standard correction
- 4. Opens window with more GPC settings
- 5 User-selectable report configuration
- Activates slice report
 Report subsets for
- information on certain fractions
- Delay for convenient calibration of second to fourth detector
- 9. Choice of baseline and evaluation and
- evaluation range setting 10 Mass distribution based on time or g/mol
- 11 Flow entry if 1100 Series pump is NOT used

Figure 2

The GPC settings and the More GPC Settings window display the necessary information and references for the GPC data evaluation.

among other parameters, the calibration file, the report settings and the integration/data evaluation range. These parameters are saved securely with the ChemStation method and loaded automatically for unattended operation.

With the GPC settings parameters specified, one more mouse click allows for the interactive evaluation of a polymer chromatogram or the start of a sequence for fully automatic analyses of samples, to be run overnight or over the weekend, for example.

The calibration data are stored in a calibration file. If more than one detector is in use, for example, refractive index and UV detection, it is not necessary to create separate calibration curves for each detector, since the chromatographic delay between the detectors is corrected on-line. The operator simply specifies the delay time in *Detector Configuration* of the GPC settings. Nothing else is required.

The ChemStation GPC data analysis software not only calculates the molecular weight averages and the molecular weight distributions but also provides additional rapid numeric data in up to five userdefined report subsets. These are recommended, for example, in quality control laboratories to easily determine the contribution of certain polymer fractions, such as low and high molecular weight fractions Figure 3 shows a typical report for a polymer sample. The report is user-configurable and contains, for example, information on the method and the sample, a graphical representation of molecular weight distribution and the numeric molecular weight results. Three subsets of molecular weight fractions at the user-defined molecular weights 10000, 280000 and 780000 are reported:

- Subset 1: for molecular weights up to 10000 D: 0.16 % contribution to the polymer composition
- Subset 2: for molecular weights up to 280000 D: 80.76% contribution to the polymer composition
- Subset 3: for molecular weights up to 780000 D: 100% contribution to the polymer composition

Report subsets can be also specified for the determination of molar masses at given values of the cumulative distribution.





Calibration

Calibration in GPC-SEC requires the injection of narrow distributed polymer standards of known molecular weight. The calibration curve is then obtained by calculating a graph of the standards' elution volumes versus the logarithm of the molecular weights. In the past it was necessary to enter the elution volumes manually into the calibration table. This process was tedious and error prone. With the Agilent ChemStation GPC data analysis software the calibration data are transferred conveniently and error-free directly from the baseline-corrected chromatogram, the elugram, and imported data sheets by mouse-clicks into the calibration table.

Figure 4 shows the elugram of a mixture of four polystyrene standards. By clicking at the x-axis the *Add to calibration* window is inserted.

This box contains

- the elution volume of the corresponding standard peak (as automatically obtained by the software),
- the molecular weight of the four standards (as userspecified in the sample editor), and
- the sample name (as user-speci fied in the sample table).

This is all the information needed which is automatically inserted into the calibration table by simply clicking *Add to calibration*.





The software offers a wide variety of curve fit functions – linear, 2nd up to 7th order and three proprietary ones – PSS 3, PSS 5 and PSS 7.

International GPC-SEC standard procedures require thorough checking on how well the calculated calibration curve fits the experimental data. (Refer to the chapter on compliance with international GPC-SEC standards later in this note). Therefore, the relative deviation is listed in the calibration table for each calibration point. The percentage of deviation for each calibration point or the differentiation can also be displayed.

Besides the most frequently used narrow standard calibration the user can also select:

- broad standard calibration based on papers of Mahadi¹, Weiss ² and Mori³,
- integral calibration, and
- universal calibration developed by Benoit⁴.

Optimization of evaluation parameters

For maximum flexibility the GPC data analysis software offers three possibilities to set baseline and integration (evaluation) range:

- Interactively in the top level of the GPC data analysis software (figure 5) by moving the red arrows with the mouse in the raw data and in the elugram window to the desired position, or with fixed molecular weight based settings for the distribution range. The operator simply enters the upper and lower limit of the molecular weight range.
- Interactively and automatically with the ChemStation Enhanced Integrator. The user simply specifies the integration events. The correctness of the integration events settings is user-controlled with the integration start and end marks.
- With fixed, time-based settings for baseline and integration range. In the More Settings window the operator specifies (figure 2) the start and end times of the baseline and the integration range.

Automated and unattended analysis

When calibration is performed the samples can be analyzed either interactively or completely automatically. Automation not only means acquiring the chromatographic raw data., but also obtaining GPC data analysis, reporting and recalibration. The information on recalibration standards and the unknown samples is entered into a standard ChemStation sequence



Arrows to set mass distribution range

5. Moveable arrows for baseline setting

2 3. Mass distribution and molecular weight results

Figure 5



| Line | Vial | Sample Name | Method Name | Inj/Vial | Sample Type | Cal Level | Update RF | Update RT | Interval | Sample Amount |
|--|------|-------------|-------------|----------|-------------|-----------|-----------|-----------|----------|---------------|
| 1 | 1 | pss white | GPC_STAN | 1 | Calibration | 1 | No Update | Replace | | |
| 2 | 2 | pss green | GPC_STAN | 1 | Calibration | 1 | No Update | Replace | | |
| 3 | 3 | pss red | GPC_STAN | 1 | Calibration | 1 | No Update | Replace | | |
| 4 | 49 | SANvlpd | GPC_SAM | 4 | Sample | | | | | |
| Replace original retention times/elution volumes of standard | | | | | | | | | | |

Figure 6

Sequence table for easy and complete automation including recalibration of the GPC calibration curve

table. Figure 6 shows a sequence table which provides for one injection each from three calibration mixtures (vials 1-3), replaces the original elution volumes in the calibration table with the new ones and performs the analysis including GPC data evaluation and reporting for the unknown polymer samples (vials 49).

Simultaneous HPLC and GPC report of a single analysis

Besides the molecular weight data, polymer chemists often need to know the concentration of monomers and oligomers. The Agilent ChemStation with the GPC data analysis software obtains both data automatically. The user receives a standard HPLC report with the information about the concentrations and at the same time a GPC report with all molecular weight averages and the molecular weight distribution (refer to figure 3). Simultaneous reporting saves time, sample and often expensive mobile phase.

Search Sample

Polymer laboratories typically have to analyze a large number of samples with different chemistry and from different customers. With the Agilent ChemStation GPC data analysis software it is no longer necessary to remember which sample came from which customer and was measured with which eluent. A query window allows to easily retrieve raw data and reconstruct results. All you have to do is to set up the *Search* Samples window as shown in figure 7 and all possible files are displayed.



Figure 7

Search Samples window for fast and convenient data file search

Compliance with international GPC Standards: ASTM D 5296-97, ISO/EN 13885 and DIN55672 GPC standards

The Agilent ChemStation GPC data analysis software enables compliance with national and international GPC standards such as ASTM D 5296-97, ISO/EN 13885 and DIN55672. They describe conditions for the determination of the molecular weight averages and the molecular-mass distributions of polymers by GPC-SEC. The goal of the standardization is to obtain comparable, precise and accurate molecular weight data determined in different labs. The important chapters on software deal with A. Calibration

- B. Data acquisition and evaluation
- C. Reporting

The ISO/EN and DIN standards are generally more specific than the ASTM standard although they are in close relationship with each other.

A. International standards concerning Calibration

Least square fits are frequently used to quickly check the quality of calibration curves, such as the chi square value. This is possible with the Agilent GPC data analysis software (figure 8). Since this yields only an average value for the whole curve there is a risk that parts of the calibration with bad fit may be overseen. To check how well the calibration curve fits the experimental data the ISO and the DIN standards require the relative deviation for each calibration point (figure 8). This is given by

Mp, calibration value - Mp, calculated $\times 100$ Mp, calibration value

and shall be plotted against the elution volume. From this graph it should be possible to assess whether the positive or negative deviations are random along the elution volume axis. Calibrationcurve fits, which exhibit trends in the deviation plot over particular elution ranges, are unsuitable. Figure 9 shows an example. If such distributions of residuals cannot be improved with the regression models available in a laboratory, it can be expected that the results contain greater errors and shall be stated in the test report.

Figure 10 shows the calibration curve and the deviation points for a 7th order fit. The random distribution of the black points indicates the superiority of that fit. The test for the distribution of residuals is not suited for calibration curves obtained by methods in which the measured points and those on the calibration curve automatically coincide, as is the case with a connected series of straight lines and with uncompensated spline algorithms ^{6,9}.



Figure 8 Important information displayed about calibration curve



Figure 9

Calibration curve (grey balls) for polystyrene standards obtained with an insufficient 3rd order fit deviation points (black balls) not randomly distributed



Figure 10

Good calibration curve (grey balls) for polystyrene standards obtained with a 7th order fit (deviation points (black balls) randomly distributed

The ISO and the DIN standards specify that the software should allow differentiating the equation describing the calibration curve. This is explained with the evaluation of the chromatograms, which involves their conversion into differential distribution curves and the reciprocal of the first derivative 1,2.

Differentiation is possible with linear and 2nd to xth order curve fits but not with a point-to-point calibration curve fit which should therefore be avoided.

Although not a requirement of international GPC standards stateof-the-art software packages should not only contain fit functions as linear and 2nd to 7th order ones but also more sophisticated functions with proven advantages. The Agilent ChemStation GPC data analysis software therefore offers in addition the functions PSS 3, PSS 5 and PSS 7. These special calibration functions can drastically increase the accuracy of molecular weight data as tested with polymer standards of known molecular weight ⁵.

Molar mass deviations of up to 100 % are possible using widely applied 3rd order polynomial functions to represent calibration data. Using the PSS calibration function differences in molecular weights are less than 5 % for weight average molecular weights. The error of the GPC method in a whole is generally estimated to be 5 % for weight average and about 10 % for number average molecular weights.

| Polystyrene Standard | PSS Calib | ration Fit | Results calculated with 3rd order polynomial | | | | |
|-------------------------|-----------|--------------------------------|---|-----------|--------------------------------|-----------------|--|
| Mw | Mw | M _w /M _n | ΔM _w | Mw | M _w /M _n | ∆M _w | |
| 985,000 | 1,006,000 | 1.05 | +2% | 1,507,000 | 1.06 | +53% | |
| 325,000 | 316,000 | 1.02 | -3% | 393,000 | 1.04 | +21% | |
| 98,000 | 96,300 | 1.05 | -2% | 84,500 | 1.07 | -16% | |
| 34,000 | 31,500 | 1.05 | +3% | 26,400 | 1.05 | -29% | |
| 10,000 | 9,830 | 1.09 | -2% | 7,800 | 1.06 | -28% | |
| 8,100 | 8,080 | 1.08 | 0% | 6,650 | 1.04 | -22% | |

Table 1

Comparison of molecular weights calculated by PSS calibration function and 3rd order polynomial function

Table 1 shows the differences in calculated molar masses for polystyrene standards with narrow molecular weight distribution in relation to different calibration functions.

B. International standards concerning data acquisition and evaluation

Here the standards specify conditions for

- determination of the baseline,
- correction of the measured values and of the net chromatogram,
- evaluation limit,
- equations for the calculation of the molecular weight averages, and
- equations for the determination the distribution curves.

These requirements are typically fulfilled by commercially available GPC-SEC software with the possible exception of the correct calculation of the differential molecular weight distribution curve. This plot of relative frequency of molecules W versus log10M requires the use of the following equation to calculate W from the net chromatogram with the abscissa Ve(elution volume) ^{6,7,8}.

$$W(\log_{10} M_i) = (-1) \times \frac{H_i}{\sum_{j=1}^{j=n} H_j} \times \left(\frac{\mathrm{d}V_{\theta}}{\mathrm{d}\log_{10} M}\right)_i$$
$$= (-1) \times \frac{H_i}{\sum_{j=1}^{j=n} H_j} \times \left(\frac{\mathrm{d}r_{\mathrm{R}}}{\mathrm{d}\log_{10} M}\right)$$

The normalized net chromatogram height is multiplied by the negative reciprocal of the first derivative of the calibration curve. If this multiplication is not done the molecular weight distribution will not be corrected for the performance of the GPC apparatus and the column(s). The result is an incorrect molecular weight distribution. The molecular weight distributions for the same polymer obtained with different systems will be different!

C. International standards regarding reporting

Round robin tests of an European study resulted in lab to lab differences for M_n of \pm 16 % and for M_w of \pm 9 % ⁹. Similar data were reported from a Japanese round robin test ¹⁰.

If details of the measuring conditions are known the chance to predict and explain such big divergences is better. Therefore, the ISO/EN and the DIN standards demand a very detailed report. The full list of requirements is about three pages long⁶ and cannot be discussed in detail here.

The benefit of the Agilent Chem-Station GPC data analysis software is that the user can fully specify the report according to his needs. For example, he may obtain a comprehensive 1-page report with the most important analysis and sample parameters, the molecular weight distribution and the molecular weight data (figure 3) but also a very detailed report, consisting of several pages. Such a detailed report may include the 1-page GPC report, and in addition a self-configured header, sample information, instrument conditions, logbook, chromato-gram and quantitative results (for example, of monomers).

Another possibility is *Area Slice* reporting which shows tabular information for each acquired slice on slice number, retention



Figure 11

Area slice report printed with molecular weight results and calibration curve data

time, molecular weight, logarithm of molecular weight, and its area derived from elugram, differential and cumulative molar mass distribution (figure 11). This report type is recommended for users who want to implement customized calculations, for example with spreadsheet applications.

The extensive reporting capabilities are obtained by taking full advantage of the standard Chem-Station's and the GPC data analysis' reporting capabilities. The different report types can be printed on paper, displayed on screen and saved as an electronic file.

Qualification procedures dedicated to GPC-SEC Analysis— Installation Verification, System Verification, System Test, Operational Qualification/Performance Verification (OQ/PV)

An increasing number of analytical polymer laboratories have to work under GLP/ISO 9000 regulated conditions. For these laboratories it is imperative to prove that the software is factory-validated and that the instrumentation passes regular qualification tests.

Most instrument manufacturers nowadays provide either manual or automated procedures, which show that hard- and software operates according to specifications. These tests are developed for typical HPLC analyses and are often not suited for the qualification of GPC-SEC systems.

The Agilent 1100 Series GPC-SEC system comprises the following qualification features dedicated to GPC-SEC:

- factory validated software,
- installation verification
- GPC system verification
- GPC system performance test
- GPC operational qualification/ performance verification

These qualification procedures are in addition to a most complete offering of qualification solutions developed for general HPLC. For an overview on the available solutions for general HPLC refer to the brochure *Five steps to fast, costeffective and successful validation* Agilent publication number 5968-9104E.

| shlwapi.dll | | | | Version 4.72 PLATFORM_W |
|-----------------|---------------------|---------|-----------|--------------------------|
| shell32.dll | | | | Version 4.00 or older |
| cometl32.dll | | | | Version 4.72 PLATFORM_N1 |
| calibration.cfg | 03.11.2000 14:48:19 | 15283 | 663478 | passed |
| gpc_top.mac | 23.08.2000 11:46:20 | 3156 | 213424 | passed |
| gpc_menu.mac | 26.09.2000 13:18:06 | 33364 | 2272479 | passed |
| gpc_prn.mac | 26.09.2000 12:57:18 | 14344 | 1051297 | passed |
| gpc.mac | 24.08.2000 09:51:24 | 25396 | 1763797 | passed |
| gpcaddon.pdf | 02.11.2000 21:17:27 | 727655 | 82683809 | passed |
| wingpc6.key | 21.06.1999 08:29:00 | 171 | 13929 | passed |
| wingpc6.exe | 01.11.2000 12:25:19 | 3199032 | 338568944 | passed |
| wingpc6.tci | 01.11.2000 12:27:28 | 119090 | 1626291 | passed |
| lang.dll | 01.11.2000 12:24:08 | 102400 | 9334011 | passed |
| hpgpc.exe | 31.10.2000 18:52:19 | 131072 | 11639685 | passed |
| gpc.hlp | 02.11.2000 20:49:14 | 1150323 | 179620896 | passed |
| gpc.cnt | 02.11.2000 20:46:11 | 8796 | 807176 | passed |
| gpc.hpj | 02.11.2000 20:46:11 | 8148 | 725095 | passed |
| bta_zip.exe | 30.03.2000 09:21:01 | 1981434 | 247860893 | passed |
| btagentbar.cnt | 28.07.2000 13:14:28 | 510 | 46480 | passed |
| btagentbar.hlp | 15.03.2000 05:55:13 | 391368 | 65139057 | passed |
| cm32cr4.dll | 16.06.2000 12:14:14 | 86016 | 8048685 | passed |
| cm32ct7.dll | 23.05.2000 14:15:07 | 350208 | 34509706 | passed |
| cm32dw5.dll | 30.05.2000 15:44:23 | 266240 | 26689986 | passed |
| cm3217.dll | 13.06.2000 09:32:02 | 1395712 | 150449717 | passed |
| cm32l700.lng | 16.06.2000 10:21:27 | 260608 | 19133932 | passed |
| cm32l701.lng | 16.06.2000 10:21:29 | 251904 | 18598909 | passed |
| cm32l7ex.llx | 16.06.2000 12:01:10 | 362496 | 34208261 | passed |
| cm32pr3.dll | 05.06.2000 08:02:21 | 110592 | 10632640 | passed |
| cm32ut5.dll | 23.05.2000 14:16:27 | 118784 | 11060632 | passed |

Figure 12

InstVeri-window proofing correct installation of the GPC-SEC software

A. Installation verification

Regulated laboratories periodically have to provide evidence that the software is installed correctly. Agilent ChemStation data analysis software features installation verification as a separate function, executable with one mouse click.

To verify correct installation of the core components the *Instveri.exe* utility is performed. Figure 12 shows the output if the installation is correct.

B. System verification

To prove that the software calculates correctly a data and calibration file obtainded from an exactly known Flory-Schultz distribution-(both provided as a protected part of the program) will be processed and a report will be automatically generated as a print-out. Figure 13 shows a report for a system verification test passed successfully. The GPC-SEC raw data from the known sample are processed in exactly the same way as data, which will be acquired by the Agilent ChemStation. This ensures that not only the final calculations are verified but also the complete processing path.





Report for a successfully passed system verification test

C. GPC system performance

The most important property of a GPC equipment is the quality of the column or column combination. With a mouse click the Agilent 1100 Series GPC data analysis software calculates the parameters listed on the right. The report shown in figure 14 is displayed. This enables a fast and comfortable control of the column(s) performance according to international GPC standards ASTMD5296, ISO 13885, DIN 55672).

| System Test | × |
|----------------------|---------|
| Column length [cm] : | 60.00 |
| Column ID [cm] : | 0.75 |
| System Test (DIN 5 | 5672) |
| Plate Count [1/m]: | 58450 |
| Asymetry [-]: | 0.747 |
| Resolution [-]: | 3.743 |
| Efficiency [cm]: | 7.002 |
| | |
| Calculate | rint OK |

Figure 14 Example for a system performance report The parameters calculated by the system test utility are:

- Plate count
- Asymmetry

$$A = \frac{W_1}{W_r}$$

where w is peak width on left or right side, measured at 10% of full peak height

$$R_{sp} = \frac{R_s}{\lg (M_1/M_2)} = \frac{0.579}{\sigma \times D}$$

• Resolution

with D of slope of the calibration curve

$$R_{s} = \frac{V_{2} - V_{1}}{(\sigma_{1} + \sigma_{2})} = \frac{\lg (M_{1}/M_{2})}{2 \times D \times (\sigma_{1} + \sigma_{2})}$$

R_{sp} specifies the quality of two peaks, whose molecular weight differs by a factor of 10, limit => 1.7 (ASTM D5296)

Efficieny =
$$\frac{V_e (M) - V_e (10 \times M)}{\text{area of column cross section}}$$

• Efficiency

ISO 13885 and DIN 55672 specify > 6 cm, that is, the separation distance between both peaks must be a minimum of 6 cm.

D. GPC Operational Qualification/Performance verification (OQ/PV)

Operational qualification is the process of demonstrating that an instrument will function according to the operational specification in the selected environment. The tests have to be performed by the user on a regular basis. In general, users should select time intervals so the probability is high that all parameters still are within the operational specifications.

The 1100 Series GPC system concept includes:

- automated OQ/PV of the GPC data analysis software which is performed by running the system verification test (refer to *B. System Verification*), and
- OQ/PV of equipment hardware and software. This includes rigorous performance testing of the instrument using the certified broad test samples and the step-by-step procedure described in the instruction manual of the start-up kits "*Getting Ready for GPC-SEC Analysis*" (Agilent part number 5064-8451 for organic eluents, 5064-8252 for aqueous eluents). The factory recommended acceptance criteria can be modified by the operator.

Summary

The Agilent GPC data analysis software for the HPLC ChemStation adds tremendous value to the Agilent 1100 Series GPC system. It is built on the well-proven standard Agilent ChemStation with a flexible data acquisition engine. The ability to use various calibration procedures to set up a data evaluation method, and the choice of various automated and interactive procedures for data processing is of great convenience to the user. Data integrity is ensured by following the automated procedures for software and instrument qualification built into the software. This is a truly easy and costeffective tool for precise GPC-SEC analysis in routine polymer laboratories.

References

1. H.K. Mahadi, K.F. O'Driscoll, J. Appl. Polym. Sci., 21, 1283 (**1977**)

2. A.R. Weiss, E. Cohn-Ginsberg, J. Polym. Sci. Part B, 7, 379 (**1969**)

3. S. Mori, Anal. Chem. 53, 1813 (**1981**)

4.

H. Benoit, Z. Grubisic, P. Rempp, D. Decker, J.-G. Zilliox, J. Chin.the. Phys., 63, 1507 (**1966**)

5.

C. Johann, P. Kilz,, Proc. Mol Mass, p. 16-18, (**1989**)

6.

ISO 13885-1 (**1998**), Binders for Paints and Varnishes - Gel Chromatography - Part 1: Tetrahydrofuran as eluent

7.

DIN 55672-1(**1995**), Gelpermeationschromatographie(GPC), Teil 1: Tetrahydrofuran als Elutionsmittel

8.

ASTM D 5295-97, Standard Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size Exclusion Chromatography (**1997**)

9.

R.J. Bruessau, Maromol. Symp. 110, 15-32 (**1996**)

10.

Hiroshi Aida et al., Kobunshi Ronbunshu, 48(8), 507-515 (**1991**)

www.agilent.com/chem

Copyright © 2000 Agilent Technologies All Rights Reserved. Reproduction, adaptation or translation without prior written permission is prohibited, except as allowed under the copyright laws.

Published March 1, 2001 Publication Number 5988-2265EN



Agilent Technologies