

Agilent Polymer Standards for GPC/SEC



Agilent polymer standards

Polymer standards from Agilent are produced in a specialized manner to impart specific characteristics not found in mass-produced counterparts. The use of stringent synthesis conditions and purified reagents results in polymers of the highest purity and the narrowest molecular weight distribution available. This is an important consideration when selecting polymer reference standards.

Agilent has 40 years of experience in the synthesis and characterization of polymers, employing a wide variety of proprietary techniques. With such extensive characterization, Agilent polymer standards are ideal reference materials for use in analytical techniques that require calibration methods or routine checks of performance.

The polymers selected for the kits have been chosen to complement Agilent GPC/SEC columns, and to produce the equidistant calibration plot necessary for accurate polymer analysis by GPC/SEC.

Quality and traceability

The Agilent philosophy is based on producing the most reliable, extensively characterized polymer standards available worldwide, and on ensuring continuity of supply and reproducibility of replacement batches.

Agilent polymer standards are reference materials that have been manufactured and tested under a Quality Management System registered to ISO 9001. Each polymer standard is assigned a unique batch number and is supplied with a Certificate of Analysis that details the exact methods used and the characterization results assigned.

At Agilent we are totally committed to the quality, reliability, and accuracy of our range of polymer standards, giving the customer security in the knowledge that they have purchased the best reference materials available.

Characterization of Agilent polymer standards

Polymers consist of a distribution of many molecular species. The average molar masses (M) reported result from several possible methods of averaging the different species present. Agilent polymer standards are characterized by various well-defined methods:

- Light scattering to determine M_w
- Viscometry to determine intrinsic viscosity
- High-resolution gel permeation chromatography (GPC) to determine M_n , M_w , M_w/M_n , and M_p .

Agilent polymer standards for GPC/SEC calibration

Calibration procedure

Both conventional GPC/SEC using a single concentration detector and GPC/SEC viscometry using universal calibration rely on an initial calibration.

Old calibration curves produce increasingly inaccurate data with time. Calibration at the start and end of a sample set is best practice. At minimum, systems should be calibrated at startup, and weekly thereafter.

When constructing a GPC/SEC calibration plot, the M_p value for the standard should be used. Operating conditions for different analytical techniques vary according to the type of polymer and the molecular weight. Full details of the analysis conditions are described on the Certificate of Analysis, together with the expiration date.

Appearance

Individual polymer standards: White powder, waxy lumps, or viscous liquid according to M_w and composition, see the note below.

Agilent EasiCal: Two sets of PTFE spatulas, differentiated by the presence of a small hole, with the polymer mixture deposited on the small end.

Agilent EasiVial: Three sets of color-coded glass vials with a polymer film deposited on the inner surface. Polystyrene and PMMA EasiVials are supplied in clear vials. PEG and PEG/PEO EasiVials are supplied in amber vials.

Note: Very low molecular weight polymers are liquid or semisolid at room temperature. Similarly, many low molecular weight standards will soften or liquefy if exposed to mild heat. Liquid standards are most easily handled by glass pipette. Softer polymer standards that have aggregated may be refrigerated until solid, carefully cut with clean shears, and handled as chips.

General preparation guidelines

The use of freshly prepared polymer standards is recommended. Standards should be dissolved in the same batch of mobile phase being run in the columns. Always allow at least three hours for molecular weight standards < 500,000 g/mol to fully dissolve. If an internal flow rate standard is being used, it needs to be monodisperse, eluted after the separation limit, and only added to the polymer standard solution. An analytically pure small molecule is generally recommended.

High molecular weight polymers (> 500,000 g/mol) are also more likely to suffer from mechanical or thermal degradation during sample preparation. These polymer standards should be allowed to swell and dissolve slowly, for at least 24 hours, with only gentle stirring. The exact system conditions of concentration, injection loop size, and so forth, will vary per system setup.

Preparation of Agilent EasiVial standards

Prepare EasiVial standards by adding solvent to one of each color-coded containers. The concentration of the standards will depend on the dn/dc for the solvent system in use and the relative sensitivity of the detectors.

Table 1. Recommended solvent volumes.

Solvent System	2 mL Vials	4 mL Vials
Polystyrene in THF ($dn/dc = 0.185 \text{ mL/g}$)	1 mL	2 mL
Polystyrene in TCB ($dn/dc = 0.053 \text{ mL/g}$)	0.5 mL	1 mL
PMMA in THF ($dn/dc = 0.085 \text{ mL/g}$)	1 mL	2 mL
PEG/PEO in water ($dn/dc = 0.136 \text{ mL/g}$)	1 mL	2 mL

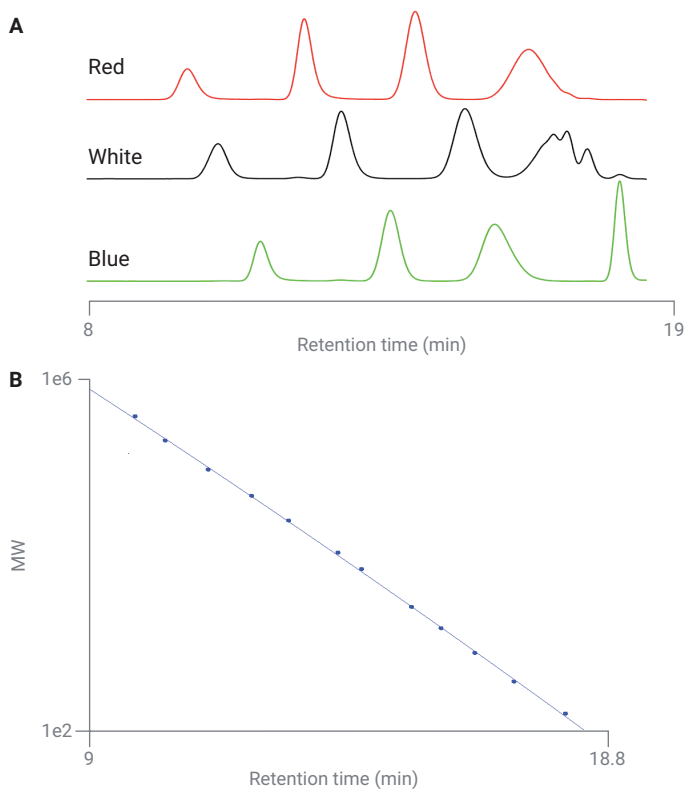


Figure 1. Typical PS-M EasiVial chromatograms and calibration plot.

Preparation of Agilent EasiCal standards

Prepare EasiCal standards by filling two vials with solvent, removing one of each type of spatula from the package, and stirring one spatula into either vial.

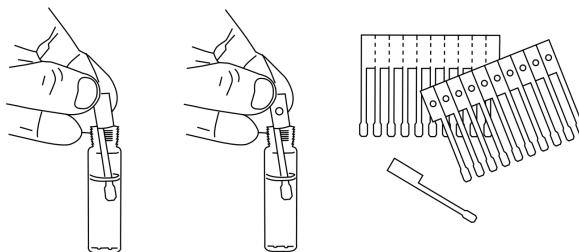


Figure 2. Preparation of Agilent EasiCal standards.

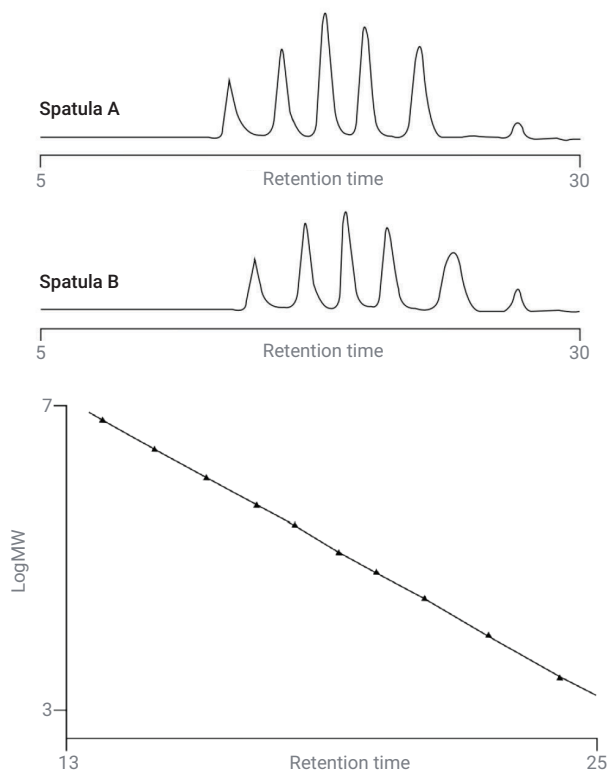


Figure 3. Typical PS-1 EasiCal chromatograms and calibration plot.

Preparation of individually bottled standards

Weigh out individual standards and dissolve them in the eluent according to the recommended concentrations in Table 2. For accurate results, follow instructions given in the "General preparation guidelines" section.

Table 2. Recommended concentrations.

Peak Molecular Weight (M_p , g/mol)	Concentration (mg/mL)
< 5,000	< 1.0
5,000 to 25,000	< 0.5
25,000 to 400,000	< 0.25
400,000 to 2,000,000	< 0.20
> 2,000,000	< 0.05

Table 3. Example of mg/mL or a 0.1% wt/vol solution.

Mass (g)	Volume (mL)
0.001	1.0
0.01	10

The size of the injection loop also determines the quantity of polymer being injected onto the column. Refer to the GPC/SEC columns user guide (publication number [5991-3792EN](#)) for recommended values.

Low molecular weight standards resolve with multiple oligomeric peaks. The highest peak is used for the M_p of the standard.

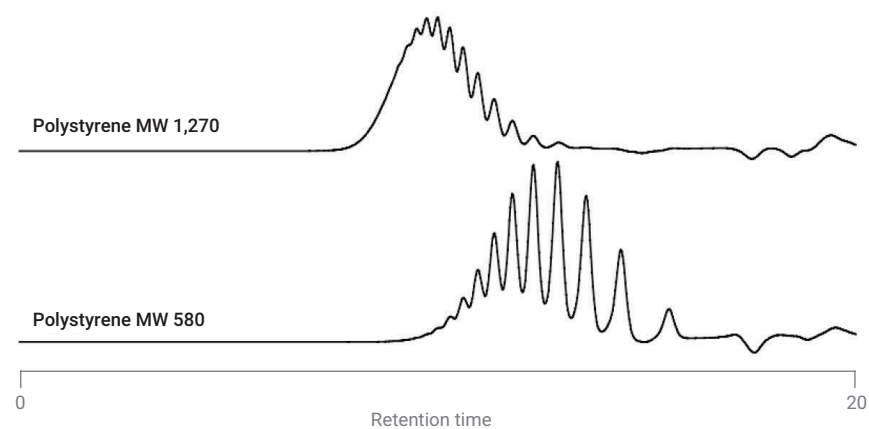


Figure 4. Typical chromatograms of low molecular weight standards showing oligomeric detail.

Summary

- Calibration curves lose accuracy over time, causing error. Calibrate before and after critical sample sets. For standard work, calibration at startup and weekly thereafter is adequate.
- Verify that the molecular weight range of the polymer standards covers the complete resolving range of the columns listed in the selection guide.
- Accurately prepare fresh solutions as per Table 1, and allow the correct length of time, given the molecular weight, for complete dissolution.
- It is possible to generate a custom mixture of individual standards, provided they do not coelute. The molecular weights should vary by an order of magnitude, for example, for polystyrene: 1, 10, and 100 kDa. The convenient EasiVial and EasiCal ranges have been specially selected to avoid coelution.
- Take care to avoid overloading the column. Use Table 2 as a guide to select concentrations, minimizing the sample load on-column where possible.
- Construct your GPC/SEC calibration using the M_p values stated on the certificates.

Storage

Most standards should be stored under normal conditions (at room temperature, in a drawer or cupboard) and do not require special storage conditions. Once prepared, dilute polymer solutions are prone to degradation, and it is recommended that they are stored in a cool, dark environment for no longer than seven days.

Polyethylene oxide standards react slowly with oxygen to form peroxides along the backbone in a process that is facilitated by light. These peroxides can lead to chain cleavage. Therefore, it is recommended that polyethylene oxides be stored refrigerated under an inert gas (argon or nitrogen).

Additional resources

- GPC/SEC Standards Product Guide, *Agilent Technologies*, publication number **5990-7996EN**.
- Calibrating GPC Columns: A Guide to Best Practice, *Agilent Technologies*, publication number **5991-2720EN**.
- GPC/SEC Column User Guide, *Agilent Technologies*, publication number **5991-3792EN**.
- Polymer-to-Solvent Reference Table for GPC/SEC, *Agilent Technologies*, publication number **5991-6802EN**.
- Step-by-Step Method Development for GPC/SEC, *Agilent Technologies*, publication number **5991-7272EN**.
- A Guide to Multidetector Gel Permeation Chromatography, *Agilent Technologies*, publication number **5990-7196EN**.

Technical support can also be obtained by contacting Agilent at: <http://www.agilent.com/en-us/contact-us/page>

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