

Agilent PL-SP 260VS Sample Preparation System for Gel Permeation Chromatography

Technical Overview

Introduction

The Agilent PL-SP 260VS is a sample preparation system designed for the manual dissolution and filtration of samples prior to analysis by gel permeation chromatography (GPC). The instrument combines controlled heating across a temperature range of 30 to 260 °C (\pm 2 °C), with gentle agitation, user selectable from 85 to 230 rpm (\pm 10%). With its wide temperature range and generous speed capability, the Agilent PL-SP 260VS is ideal for a broad range of polymer types, including the most difficult of samples.



Dissolution and Filtration

The PL-SP 260VS sample preparation system allows the dissolution and filtration of samples at temperatures of up to 260 °C. With the heater/shaker unit, samples are dissolved at controlled temperatures with gentle agitation. Filtration is achieved using a unique hand held pipettor with a selection of filter magazines containing either stainless steel frits, in pore sizes of nominally 0.5 to 10 μ m, or glass fiber filters at a nominal 1 μ m porosity. The 1 μ m glass fiber filter magazine is particularly suited to the removal of fine, insoluble particulates such as carbon black. Its double layer acts as a depth filter, fit for use with even high molecular weight polymers. Figure 1 shows a successfully filtered solution of polyethylene containing carbon black.

However, during filtration, it is important that no high molecular weight fractions of the sample are retained on the filter, as this would result in errors in concentration and distortion of the molecular weight distribution.

To demonstrate the ability of the system to handle such conditions, a series of polymer solutions, also from Agilent Technologies, were prepared in THF containing a high and low molecular weight polystyrene standard. The solutions were analyzed by GPC in THF before and after filtration using the 1 μ m glass fiber filters to determine if any loss of the high molecular weight material occurred. Polystyrene standards were prepared, each containing polystyrene Mp 126,000 at 0.5 mg/mL and either polystyrene narrow standard Mp 3,900,000, Mp 7,100,000 or Mp 11,600,000 at 0.1 mg/mL low molecular weight polystyrene standard.

Figure 1 is an example overlay of chromatograms of the Mp 126,000 g/mol and Mp 3,900,000 g/mol standards before and after filtration. It can be seen that, after filtration, none of the high molecular weight material was removed from the sample. In addition, the retention times of the high molecular weight standards were unchanged. These data clearly demonstrate the suitability of the filters for even high molecular weight polymers.

Conditions

Samples	Agilent polystyrenes
Column	Agilent PLgel 10 μ m MIXED-B, 7.5 × 300 mm (p/n PL1110-6100)
Eluent	THF (stabilized)
Flow rate	1 mL/min
lnj vol	200 µL
Detector	UV, 254 nm
Sample prep	Agilent PL-SP 260VS
System	Agilent PL-GPC 50



Figure 1. Comparing overlaid chromatograms of polystyrene Mp 126,000 g/mol and Mp 3,900,000 g/mol before and after filtration on the Agilent PL-SP 260VS sample filtration system.

GPC/SEC Columns and Calibrants from Agilent

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