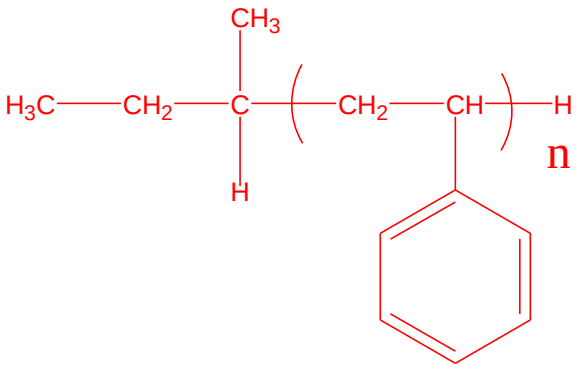
Polymer Reference Materials-Polystyrene

Introduction

Reference materials are used for calibration and performance evaluation of instruments used as part of overall quality assurance programs of polymer. These materials support the development of new measurement methods and characterize new materials. The synthesis and characterization of our polymer reference materials are achieved meticulously. Above all, the user can be assured of receiving a well-characterized and quality product.

Chemical Structure of the polymer showing end groups:



Molecular weight based reference polymers

Polymer Source offers a wide range of reference polymeric materials (RM) for both organic and aqueous phase applications. New reference polymers are added regularly. A wide range of polymers with number average molecular masses (M_n) (oligomers ***to 10 million***) have been covered.

Molecular weight values are characterized by various analytical techniques. Interfacing chromatographic methods with other analytical techniques can significantly increase the amount of information available for polymer characterization. The techniques used for characterization are: size exclusion chromatography (SEC), nuclear magnetic resonance

(NMR), intrinsic viscosity, thermal analysis, and Matrix Assisted Laser Desorption Ionization-Time of Flight-Mass Spectrometry (MALDI-TOF-MS).

Organic phase soluble	Polystyrene	Polymethyl methacrylate	Polybutadiene	Polyisoprene
Aqueous phase soluble	Polyethylene oxide (PEO)	Polyacrylic acid	Polystyrene sulfonic acid	Polystyrene sodium sulfonate

The polymer standards are available either *individually* (in the widest range of molecular weights) or *as kits* containing wide range of M_n. Care has been taken to develop standards with the narrowest molecular weight distribution to ensure reliable calibrations of the instruments or for basic research. A *Certificate of analysis* that accompanies each product provides the characterization information indicating the type of end groups and microstructure of the polymer.

Purification of Polymer samples:

Purification of the polystyrene was carried out rigorously to ensure the removal of the catalyst by following steps:

1. Dissolved the polymer in $CHCl_3$ and washed with water to remove insoluble organic catalyst as side product.
2. Polymer solution in chloroform filtered and passed through a column packed with basic Al_2O_3 .
3. Solution was concentrated on rota-evaporator
4. Concentrated solution precipitated in cold methanol.
5. Dried under vacuum for 48h at 38 ^oC. Further, dissolved in dioxane; filter, and freeze dried under vacuum.
6. Polymer was packed in a clean vial in dust free environment.

Characterization techniques

Gel Permeation or Size Exclusion Chromatography

Gel permeation chromatography (GPC) also known as size exclusion chromatography (SEC) is employed to obtain number average molecular masses (M_n) and weight average molecular weight (M_w). Both these values result to obtain polydispersity index (PDI) ($PDI=M_w/M_n$). It guides the application level of reference polymers.

SEC analysis was performed on a Varian liquid chromatograph equipped with refractive and UV light scattering detectors. Three SEC columns from Supelco (G6000-4000-2000 HXL) were used with a dual detector model 270 from Viscotek Co connected on line in series with columns. Low angle and at 90 ^{o} light scattering were used to determine absolute molecular weights of the polymer.
Average molecular weight by weight:

$$M_w = \frac{\sum w_i M_i}{\sum w_i}$$

Average molecular weight by number:

$$M_n = \frac{\sum n_i M_i}{\sum n_i}$$

where: w_i is the weight in fraction i; n_i is the molecular number in fraction i. M_i is the molecular weight of fraction i.

$$M_i = \frac{w_i}{n_i}$$

Due to some polymer chains distribution in polymer sample, weight average molecular weights (Mw) is always greater than number average molecular weights (Mn). The index of Mw/Mn determine the molecular distribution (polydispersity: PDI) is introduced.

$$PI = \frac{M_w}{M_n}$$

If PI=1, all the chain lengths are same. Usually, the sample prepared by living process is of narrow distribution character, the PI should be less than 1.15.

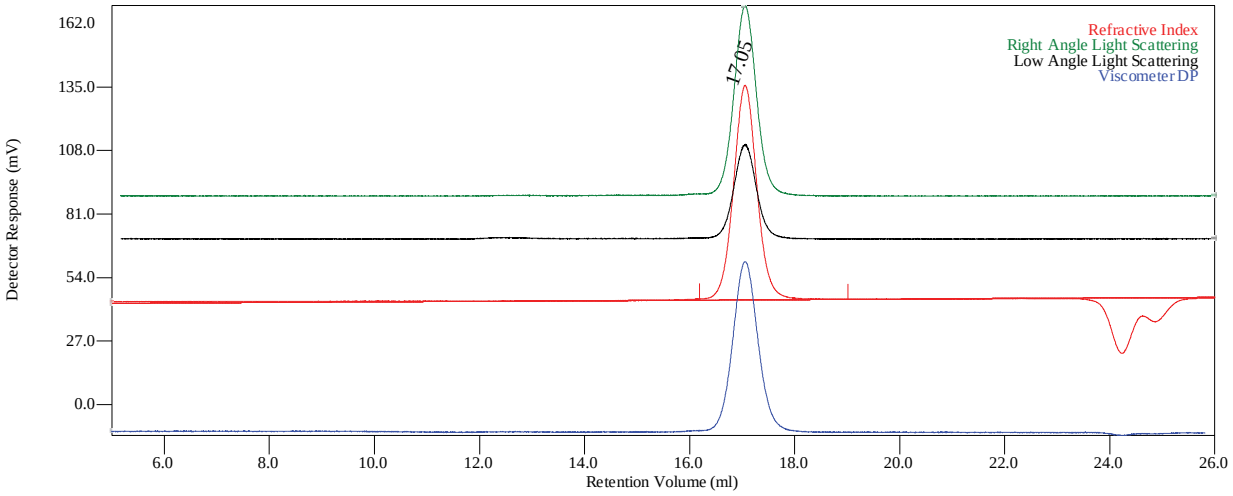
Mp is the molecular weight at peak maximum.

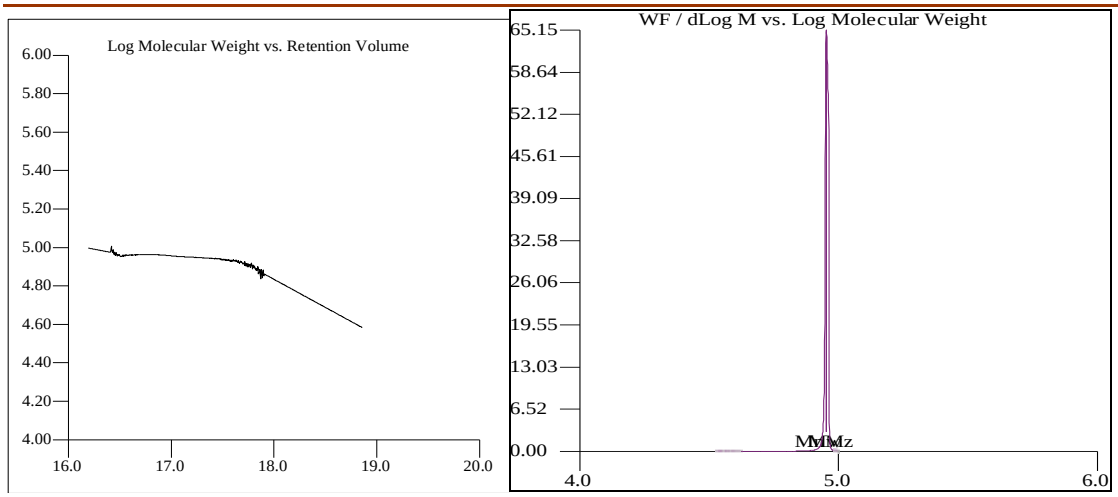
Intrinsic viscosity [η] is the related viscosity exploited to concentration = 0, which is related to solvent and temperature. The molecular shape is important to the intrinsic viscosity. When molecular weight is same, the [η] of multi-arm (four- or six-arm) polymer is much lower than that of linear one.

Radius of Gyration (Rgw): the size of macromolecule.

Infrared spectrum will give us the difference in the samples qualitatively, due to the sensibility to impurities. The three samples are chemical identical, as well as the functionality range.
It is worth to note that all the results listed here might have about ±5% deviation.

Polymers were characterized using a fully integrated GPC instrumentation (combining refractive index detectors with static light scattering detectors and viscometers) and complete software packages. A typical combined SEC of a reference polymer having M_n of 90000 dalton is shown below:



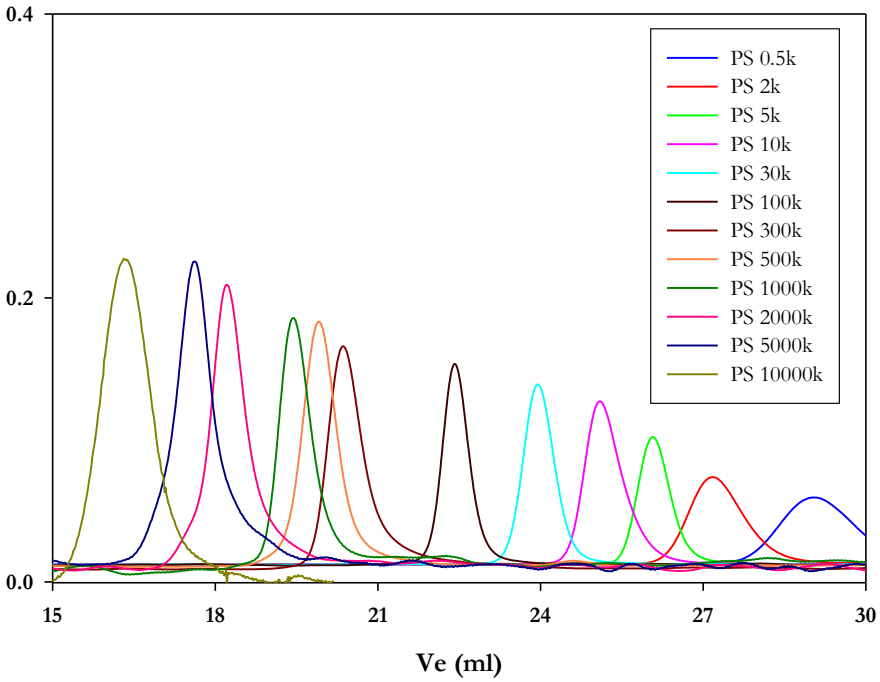


Typical SEC of Reference Materials

Polystyrene (PS)

Gel permeation chromatography (GPC) of polystyrene M_w ranging from oligomers to 10 millions are shown below.

SEC of selected polystyrene standard



Polystyrene standard samples

Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 0.5K	P8092-S	500	600	620	-	1.2
PS 1K	P8094-S	950	1140	950	-	1.2
PS 2K	P8095-S	1900	2185	2000	-	1.15
PS 5k	P2444-S	5,522	5,910	5,770	0.1176	1.07
PS 12k	P5811-S	12,800	13,800	13,300	0.2021	1.07
PS 21k	P486-S	21,000	23,000	21,000	0.2828	1.09
PS 55K	P8647-S	54,500	59,300	59,100	0.5458	1.08
PS 100k	P8070-S	99,000	105,000	100,500	0.8260	1.06
PS 235K	P5712-S	235,000	256,000	241,500	1.5880	1.09
PS 420K	P4699-S	419,000	444,000	419,500	2.4270	1.06
PS 600K	P2577-S	600,000	643,000	632,500	2.8360	1.07
PS 820K	P5654-S	820,000	878,000	880,000	3.8860	1.07
PS 5,000K	P1104-S	5,000,000	6,500,000	5,800,000		1.30
PS 6,500K	P1105-S	6,500,000	8,200,000	7,600,000		1.25
PS 7,600k	P1099-S	7,600,000	9,300,000	8,800,000	-	1.22

Prices

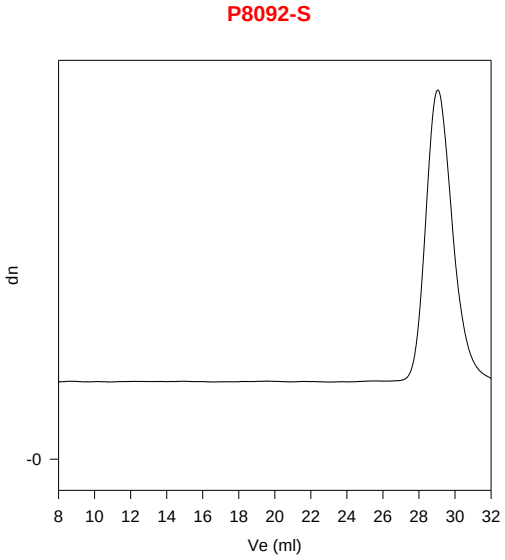
15 polystyrene reference material 500mg each US$ 1000.00

15 polystyrene reference material 1,000mg each US$ 1400.00

Characterization report for the each sample analysis:

PS: 500 lot P8092-S

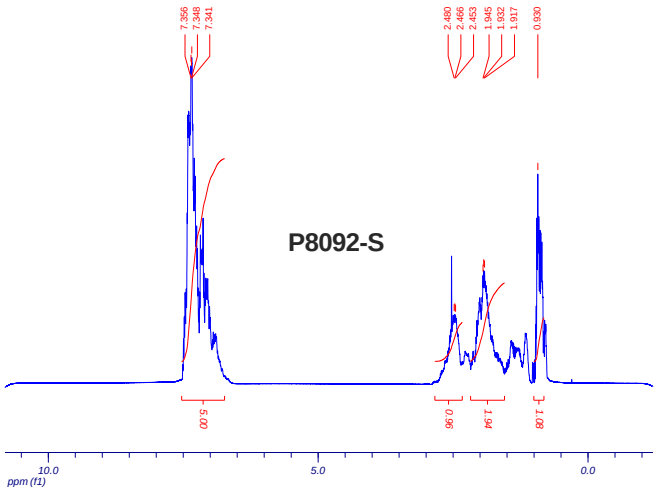
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 0.5K	P8092-S	500	600	620	-	1.2



Size Exclusion Chromatography of polystyrene

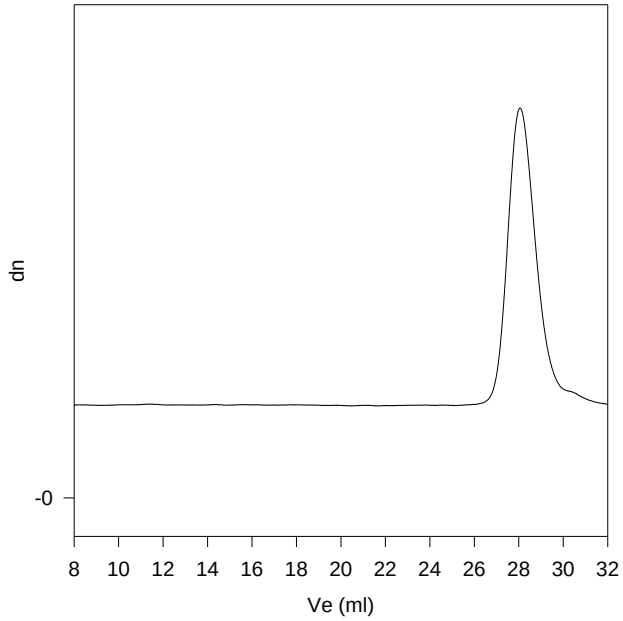
M_n=500, M_w=600, M_w/M_n=1.20

Mn by HNMR: 550



Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23^{0}C (dl/g)	M_w/M_n
PS 1K	P8094-S	950	1140	1050	-	1.2

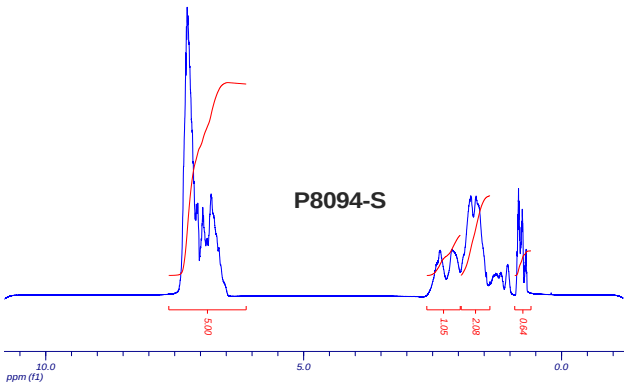
P8094-S



Size Exclusion Chromatography of polystyrene

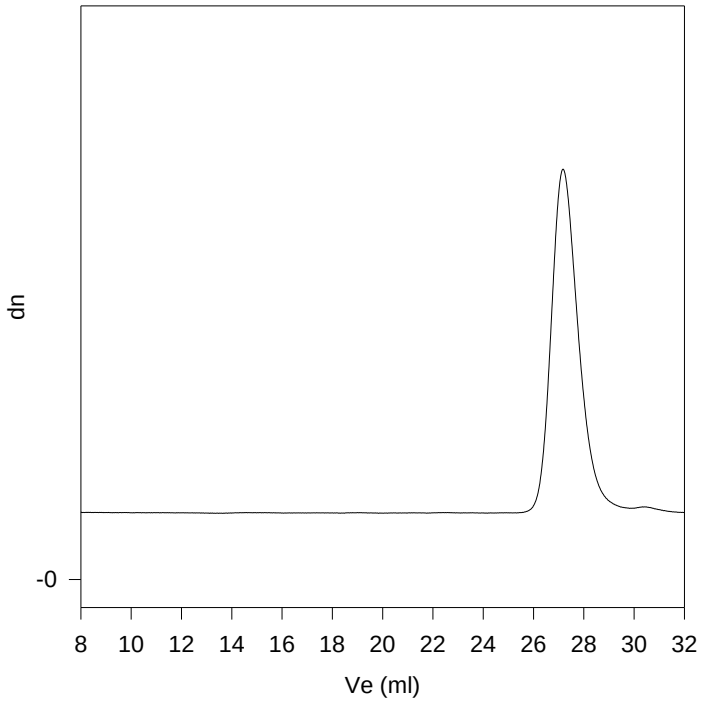
M_n=950, M_w=1400, Mp: 1050 M_w/M_n=1.20

Mn by HNMR:



Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 2K	P8095-S	1900	2185	2000	-	1.15

P8095-S

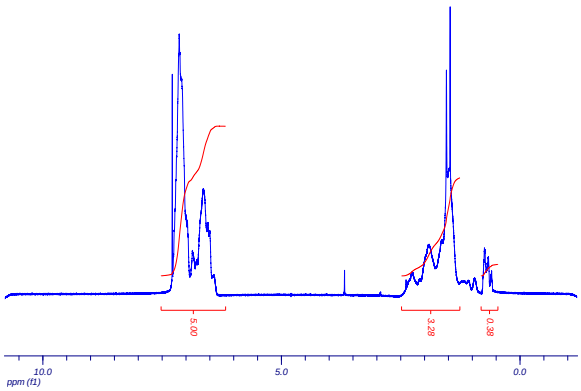


Size Exclusion Chromatography of polystyrene

M_n=1900, M_w=2200, M_w/M_n=1.19

Mn by HNMR: 1700

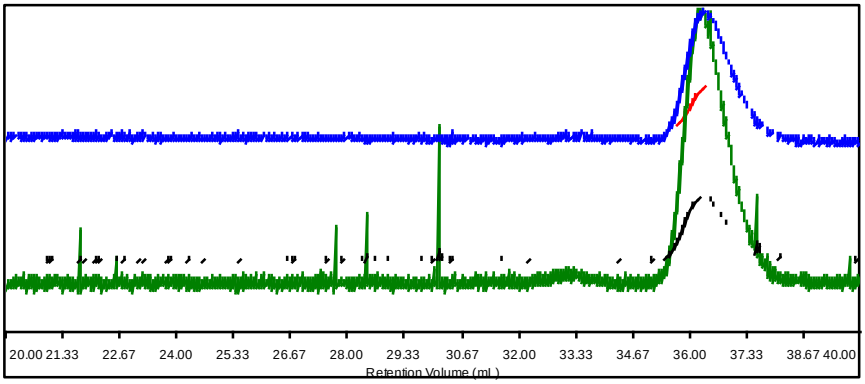
P8095-S



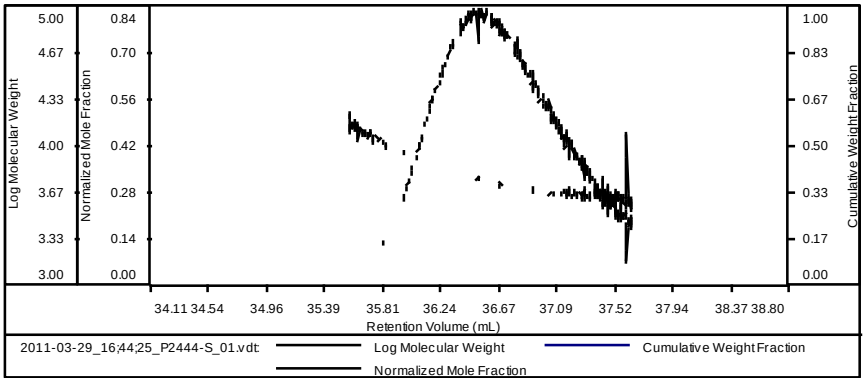
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 5k	P2444-S	5,522	5,910	5,770	0.1176	1.07

Sample ID: P2444-S

Concentration	2.1713
Sample dn/dc	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1



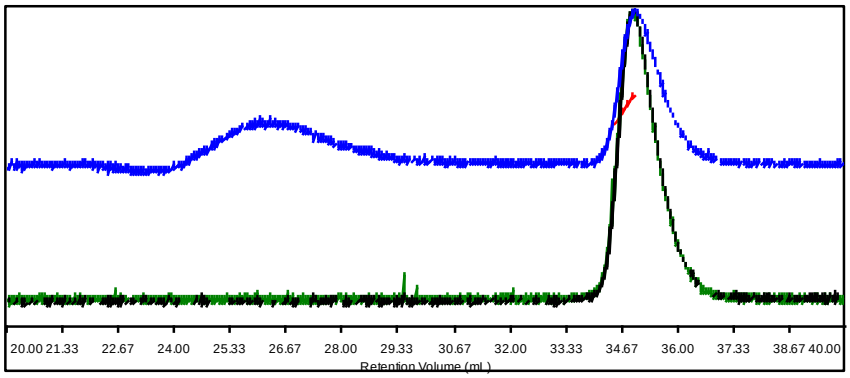
Sample	Mn	Mw	Mp	Mw/Mn	IV	Rh
2011-03-29_16;44;25_P2444-S_01.v	5,522	5,909	5,771	1.070	0.1176	2.89



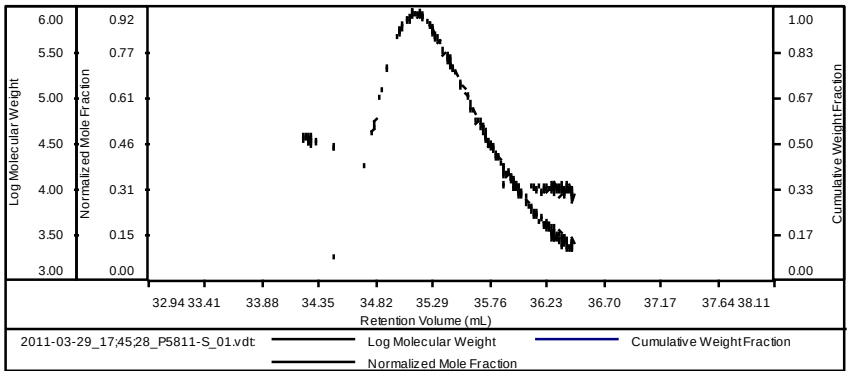
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 12k	P5811-S	12,800	13,800	13,300	0.2021	1.07

Sample ID: P5811-S

Concentration	1.6279
Sample dn/dc	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1



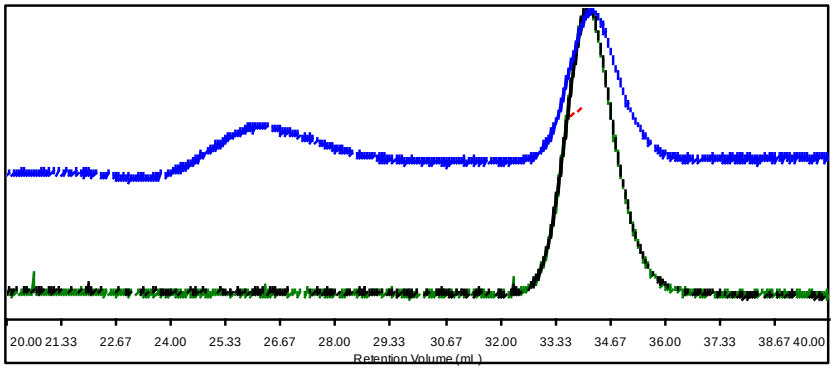
Sample	Mn	Mw	Mp	Mw/Mn	IV	Rh
2011-03-29_17;45;28_P5811-S_01.v	12,864	13,787	13,320	1.072	0.2021	4.58



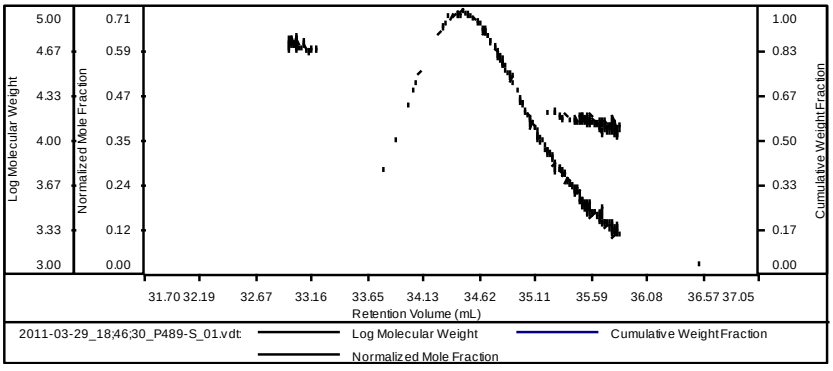
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 21k	P486-S	21,000	23,000	21,000	0.2828	1.09

Sample ID: P489-S

Concentration	1.4288
Sample dn/dc	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1



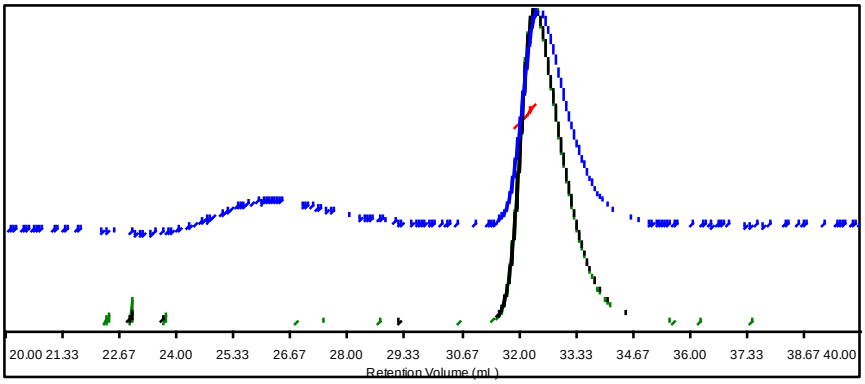
Sample	Mn	Mw	Mp	Mw/Mn	IV	Rh
2011-03-29_18;46;30_P489-S_01.vd	20,860	23,018	22,129	1.103	0.2818	6.06



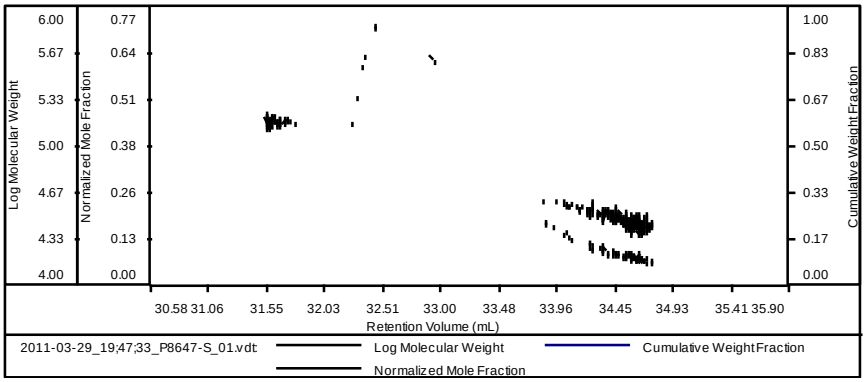
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 55K	P8647-S	54,500	59,300	59,100	0.5458	1.08

Sample ID: P8647-S

Concentration	1.4364
Sample dn/dc	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1



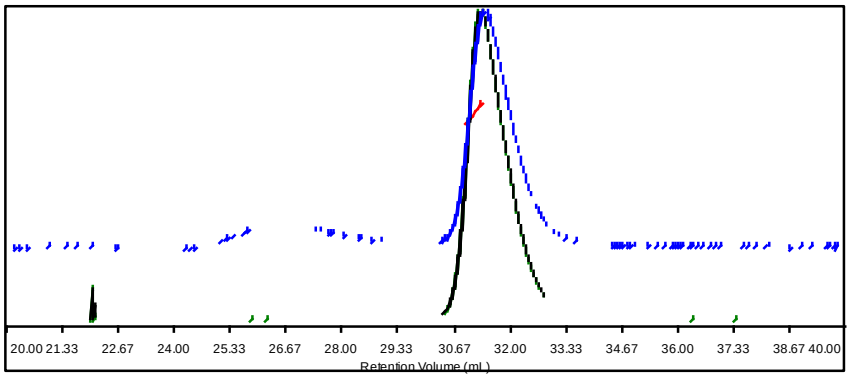
Sample	Mn	Mw	Mp	Mw/Mn	IV	Rh
2011-03-29_19;47;33_P8647-S_01.v	54,275	59,358	59,077	1.094	0.5458	10.37



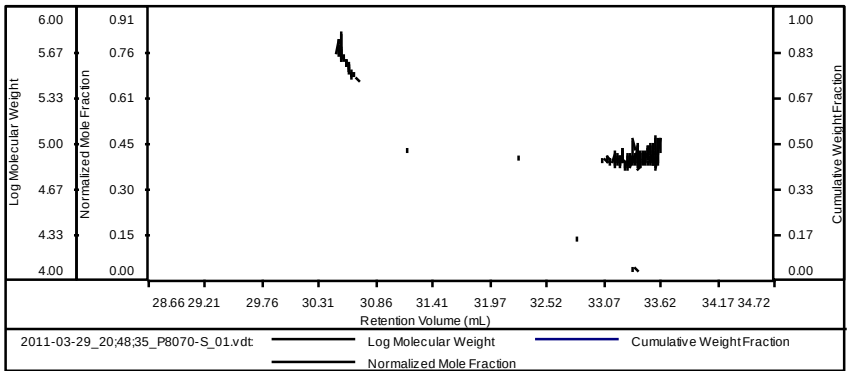
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 100k	P8070-S	99,000	105,000	100,500	0.8260	1.06

Sample ID: P8070-S

Concentration	1.3054
Sample dn/dc	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1



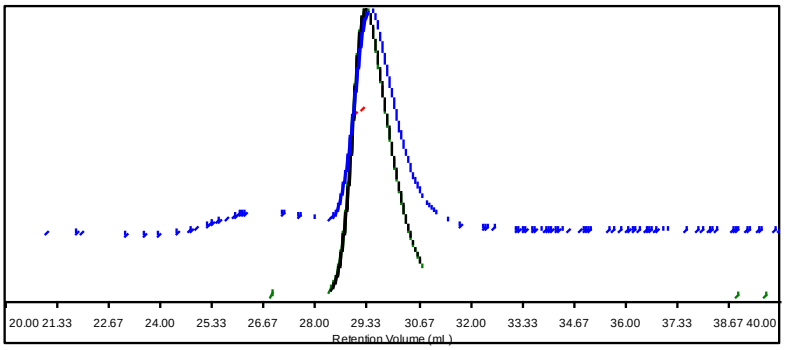
Sample	Mn	Mw	Mp	Mw/Mn	IV	Rh
2011-03-29_20;48;35_P8070-S_01.v	98,636	105,753	100,489	1.072	0.8261	14.44



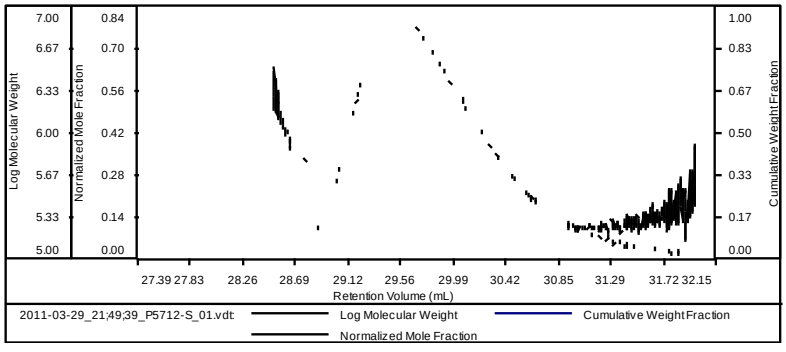
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23^{0}C (dl/g)	M_w/M_n
PS 235K	P5712-S	235,000	256,000	241,500	1.5880	1.09

Sample ID: P5712-S

Concentration	0.7563
Sample dn/dc	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1



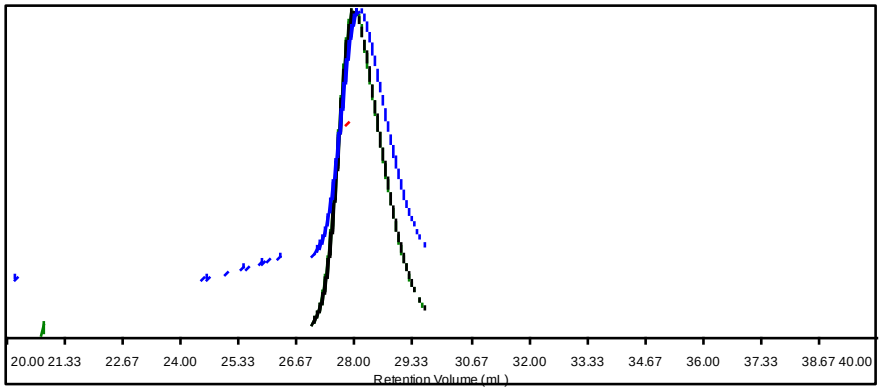
Sample	Mn	Mw	Mp	Mw/Mn	IV	Rh
2011-03-29_21;49;39_P5712-S_01.v	234,906	256,362	241,320	1.091	1.5881	24.03



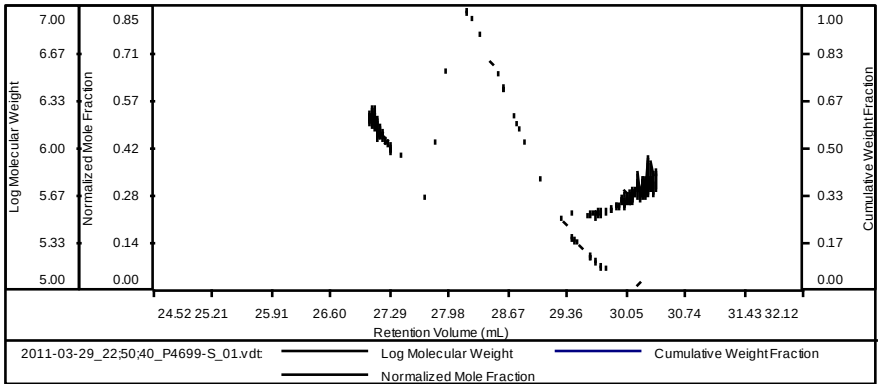
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 420K	P4699-S	419,000	444,000	419,500	2.4270	1.06

Sample ID: P4699-S

Concentration	0.7491
Sample dn/dc	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1



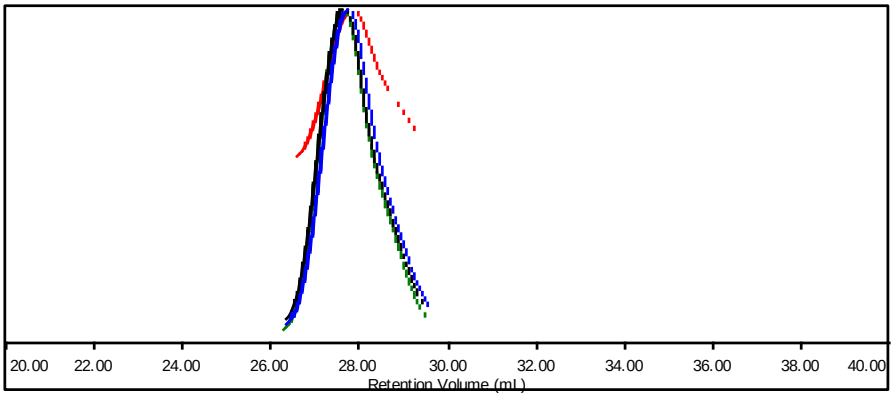
Sample	Mn	Mw	Mp	Mw/Mn	IV	Rh
2011-03-29_22;50;40_P4699-S_01.v	418,563	443,977	419,305	1.061	2.4272	33.29



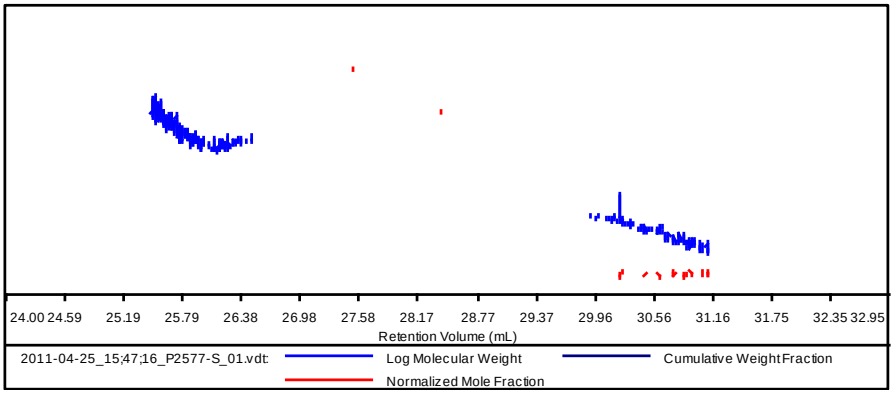
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 600K	P2577-S	600,000	643,000	632,500	2.8360	1.07

Sample ID: P2577-S

Concentration (mg/ml)	2.0014
Sample dn/dc (dl/g)	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1



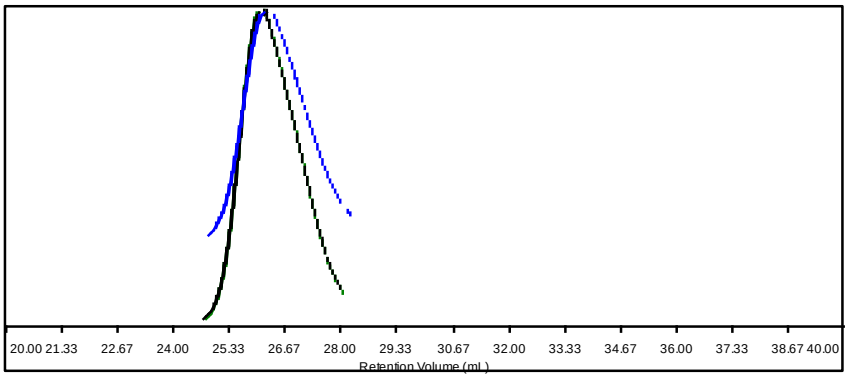
Sample	Mn (Daltons)	Mw (Daltons)	Mp (Daltons)	Mw/Mn	IV (dl/g)
2011-04-25_15;47;16_P2577-S_01.vdt	600,650	643,453	632,760	1.071	2.8360



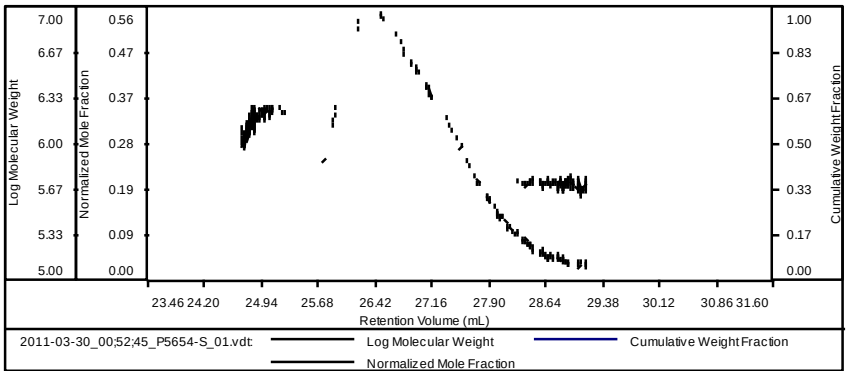
Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23°C (dl/g)	M_w/M_n
PS 820K	P5654-S	820,000	878,000	880,000	3.8860	1.07

Sample ID: P5654-S

Concentration	0.9829
Sample dn/dc	0.1850
Method File	PS80-Jan192011-0000.vcm
Column Set	3x PL 1113-6300
System	System 1

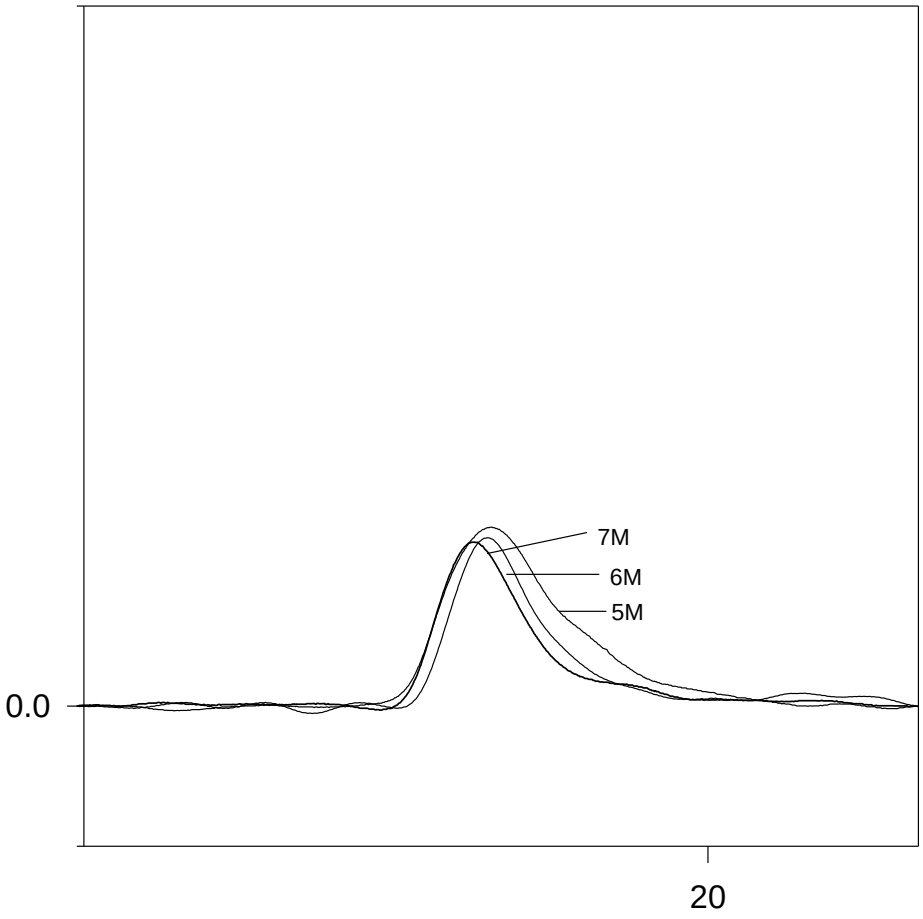


Sample	Mn	Mw	Mp	Mw/Mn	IV	Rh
2011-03-30_00;52;45_P5654-S_01.v	820,377	877,812	880,203	1.070	3.8873	49.07



Part No.	Lot No.	M_n	M_w	M_p	[η] in THF at 23^{0}C (dl/g)	M_w/M_n
PS 5,000K	P1104-S	5,000,000	6,500,000	5,800,000		1.30
PS 6,500K	P1105-S	6,500,000	8,200,000	7,600,000		1.25
PS 7,600k	P1099-S	7,600,000	9,300,000	8,800,000	-	1.22

PS of 5.0M ; 6.5M and 7.6M



—— Polystyrene, of molecular weights of 5,000,000; 6,500,000 and 7,600,000