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A COMPARATIVE STUDY OF PORASIL AND STYRAGEL AS COLUMN SUPPORTS FOR GEL PERMEATION CHROMATOGRAPHY

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SUMMARY

Molecular weight data were compared using two different column supports, Styragel and Porasil. A series of poly(styrene) and poly(vinylchloride) materials were analyzed on a Model 100 gel permeation chromatograph. The results obtained from the Porasil-packed columns were consistently lower than those obtained using the Styragel-packed columns. Data from both methods were further compared to values determined by such classical techniques as light scattering and membrane osmometry. Two experimental parameters, concentration and injection time, were included in this evaluation.

INTRODUCTION

Since the advent of the term gel permeation chromatography (GPC) in 1964 by MOORE¹, workers in the field have utilized this technique to facilitate molecular weight polymer characterization. A few GPC chemists have experimented with more effective substrates for use as GPC column packing. COOPER AND JOHNSON² have published data which proves the complete elution of polystyrene and polyisobutene on porous glass column packings. They eliminated adsorptive effects by treating the porous glass with hexamethyldisilazane. LOCHMÜLLER AND ROGERs³ compared the behavior of stable inorganic and organic species on several commonly used gel media on the basis of resolution and recovery. The gel media covered in their study included: Sephadex G-25, Sephadex LH-20 (Pharmacia, Sweden), Bio-Gel P-6 and Bio-Glas 200 (Bio-Rad Laboratories).

DE VRIES *et al.*⁴ reported the evaluation of spherical porous silica beads (Porasil) as a GPC column packing material. The pure silica nature of this material lends itself to improved chemical inertness and heat-resistance over the commonly used organic polymeric supports. According to their observations, the Porasil packing improves column efficiency at decreased flow rates and narrowed particle size distribution. The authors concluded from this investigation that the highest inherent efficiency was obtained with Porasil packings of at least 100 μ average diameter.

In an other paper, LEPAGE *et al.*⁵ described the characteristics of two types of calibration standards, polystyrene and polyvinylchloride. The polystyrenes covered

a molecular weight range of 600 to 1800000 and 6400 to 250000 in the case of the polyvinylchlorides.

The purpose of this paper is to compare data obtained using two column sets of four columns each, one packed with Styragel and the other with Porasil. Nineteen polymers of various molecular weights were analyzed on both systems and the resulting data compared and discussed.

EXPERIMENTAL

A Model 100 gel permeation chromatograph was the instrument used to carry out the analyses for the two column sets. Operating temperature was 37° using Eastman grade tetrahydrofuran as the carrier solvent. Each sample was dissolved in degassed solvent from the instrument's drain-out valve. The flow rate was regulated at a constant I cc per min.

The Styragel four column set, Set A, was obtained directly from the vendor, Waters Associates Inc. Precise column description is contained in Table I.

TABLE I

DESCRIPTION OF COLUMN SET A (STYRAGEL)

Plates/ft.
860
920
I 345
824

TABLE II

DESCRIPTION OF COLUMN SET B (PORASIL)

Туре	Mesh	Pore diameter (Å)
в	< 150	100-200
C	< 150	200-400
D	< 150	400-800
E	< 150	800-1500

The second column set, Set B, was packed with Porasil by our laboratory. Each of the four columns were hand-packed by adding the Porasil material in its original form to the column. An electric vibrator was used to aid in firm, even packing and eliminate air pockets. Table II contains the description of the Porasil packed column set, set B.

The polymers examined in this study are divided into three groups; first, the narrow distribution polystyrenes obtained from Waters Associates, Inc.; second, the broad molecular weight polystyrenes which were polymerized by our polymer synthesis group; and third, three polyvinylchloride samples distributed by ArRo Laboratories. Table III contains the absolute values obtained for use in this investigation. The data on the PS calibration standards were reported by Waters Associates and on

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the polyvinyl chloride materials by ArRo Laboratories. The \bar{M}_w values reported by ArRo Laboratories were obtained in THF at 25° by light scattering with $\lambda = 4360$ Å and a dn/dc = 0.129 ml/g. Their \bar{M}_n values were obtained using a Mechrolab 503 high speed membrane osmometer in THF at 25° using Gel Cellophane 450 membranes.

TABLE III

ABSOLUTE VALUES ESTABLISHED FOR POLYMERS USED IN THIS STUDY

Sample	Composition	M _w	\overline{M}_n	MWD	Data source
2.0K	Poly(styrene)	2 030	1 840	01.1	Pressure Chemical
5.0K	Poly(styrene)	5 000	4 600	1.09	Waters Associates Inc.
10.3K	Poly(styrene)	10 300	9 700	1.00	Waters Associates Inc.
19.8K	Poly(styrene)	19 800	19 600	10.1	Waters Associates Inc.
51 K	Poly(styrene)	51 000	49 000	1.04	Waters Associates Inc.
98.2K	Poly(styrene)	97 200	96 200	10.1	Waters Associates Inc.
160K	Poly(styrene)	173 000	164 000	1.05	Waters Associates Inc.
411K	Poly(styrene)	411 000	392 000	1.05	Waters Associates Inc.
860K	Poly(styrene)	867 000	773 000	1.12	Waters Associates Inc.
2000K	Poly(styrene)	2 145 000	1 780 000	1.20	Waters Associates Inc.
A-t	Poly(styrene)	33 000	18 200	1.83	Xerox Corporation
A-3	Poly(styrene)	126 400	51 500	2.45	Xerox Corporation
A-4	Poly(styrenc)	185 500	79 300	2.34	Xerox Corporation
A-5	Poly(styrene)	356 100	165 000	2.16	Xerox Corporation
400-2	Poly(vinyl chloride)	68 600	25 500	2.69	ArRo Laboratories
400-3	Poly(vinyl chloride)	118 000	41 000	2.88	ArRo Laboratories
400-4	Poly(vinyl chloride)	132 000	54 000	2.44	ArRo Laboratories

Using a Q factor of 25, GPC data was also obtained in THF at 25°. The column arrangement was designated 3×10^6 , 10^5 , 10^4 , and 10^3 Å porosities. The samples were prepared at 0.5% w/v concentration and injected for 120 sec at a flow rate of 1.0 ml/min.

 \bar{M}_w values for the broad distribution polystyrenes, prepared in our polymer

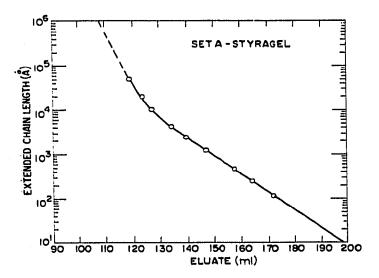


Fig. 1. GPC calibration curve, four Styragel column system, 1×10^6 , 4×10^5 , 1×10^4 , and 1×10^3 Å, 0.5 to 0.25% in THF at $1 \times$.

laboratory, were obtained from light scattering measurements at ambient temperature in MEK at a wavelength (λ) of 5460 Å. Our \overline{M}_n values were obtained using a Mechrolab Model 501 high speed membrane osmometer in toluene at 37° using Schleicher and Schuell super dense o8 deacetylated acetyl cellulose membranes⁶.

Each GPC column system was calibrated using the polystyrene standards from Waters Associates. The standards were dissolved in THF at 1/4% w/v and injected for 120 sec. The calibration curve which resulted in Set A is illustrated in Fig. 1. The same procedure was followed in calibrating Set B and the resulting calibration curve is shown in Fig. 2. All GPC calculations made use of the values from the appropriate calibration curve.

The broad distribution polymers were analyzed at various concentration levels. All of them were run at the 1/2 % w/v in THF at a 120 sec injection time. This series was analyzed on Set A and compared with those run on Set B. The nine broad distribu-

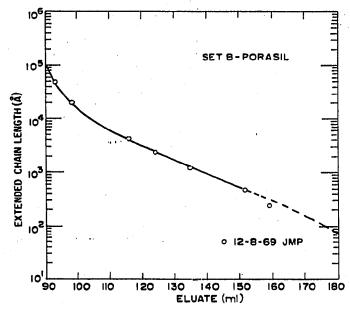


Fig. 2. GPC calibration curve, four Porasil column system, 0.25% in THF at $4 \times .$

TABLE IV

GPC	DATA	FROM	ANALYSIS	OF	\mathbf{PS}	STANDARDS

Standard PS	Set A - S	lyragel		Set $B - P$	o ra sil	
	\overline{M}_w	M _n	MWD	M w	\overline{M}_n	MWD
2.0K	3 400	3 100	1,10	7 050	6 600	1.07
5K	4 950	4 550	1.09	8 2 5 0	7 750	1.07
10.3K	11 000	9 800	1.12	13 400	12 350	80.1
19.8K	19 950	18 550	1.08	21 100	19 700	1.07
51K	53 650	49 050	1.09	52 100	47 300	1.10
98.2K	103 200	92 300	1,12	100 950	93 100	r.08
173K	157 450	137 700	1.14	174 600	157 300	1.11
411K	466 400	350 450	1.33	436 250	373 650	1,17
860K	1 007 500	666 200	1.51	931 950	662 300	1.4i
2145K	2 165 200	I 134 550	1,91	1 795 400	918 900	1.95

PORASIL AND STYRAGEL AS COLUMN_SUPPORTS FOR GPC

TABLE V

GPC DATA ON BROAD PS MATERIALS

Sample	Set A –	Set A – Styragel	ľ	Set B — Porasil	- Porasii	-												
00 1 6	I/2% -	1/2% – 2 min		1/2%	– 2 min		1/4 ^{0/} 0 – 2 min	- 2 min		1/4% – 1 min	- I min		1/8%	1/8% – 2 min	'n	1/8%	1/8% – 1 min	
	Mu ^w	\bar{M}_n	D	\overline{M}_{w}	\tilde{M}_n	Q	\overline{M}_{w} :	Мn	Q	\overline{M}_{w}	\bar{M}_n	D	<u>M</u> w	Мn	D	\overline{M}_{lr}	M_n	Q
A-1 (1) (2) (3)	34.16 34.03 34.00	17.14 17.35 17.25	66.1 96.1 70.1	32-55 31.08 30.13	21.60 18.22 17.88	1.51 1.71 1.68	31.75 31.15	20.55 18.71	1.54 1.66	29.90 31.35	20.50 20.85	1.46 1.50						
A-2 (1) (2) (3)	88.85 91.96	38.15 41.49	2.33 2.22	82.00 86.34	41.75 39.77	1.96 2.17	79.8 95.07 89.70	37.4 39.35 40.47	2.13 2.42 2.22	78.05 86.05 77.25	39.00 27.60 39.76	2.00 3.12 1.94						
A-3 (1) (2) (3)	113.3 112.47	51.2 50.69	2.21	103.35 104.14 100.11	56.95 46.66 45.72	1.81 2.23 2.19	98.40 104.83	49.50 48.38	1.99 2.17	97.12	48.79	66.1						
A-4 (I)	150.25	63.05 2.38	2.38	147.80	73.30	2.02	143.85	72.90 1.97	1-97	143-55	70.60 2.03	2.03						
A-5 (1) (2) (3)	316.90 333.99 331.26	101.40 110.18 99.73	3.12 3.03 3.32	275.15 281.65	109.55	2.51 2.60	278.10 277.40 277.13	111.80 110.96 110.39	2.49 2.50 2.51	281.62 126.44	126.44	2.23						
A-6 (I) (2) (3)	46.40 49.65 49.06	22.95 25.56 24.66	2.02 1.94 1.99	47.45	29.90	1.59	36.65	26.80	1.37	47-35 44-32	29.05 27.56	1.63 1.61	43-55 43-00	26.55 26.57	1.64 1.62	40.70 44.05 46.15	25.6 1 26.3 1 28.9 1	1.59 1.67 1.60

tion polymers were also run at 1/4 % w/w at 120 and 60 sec injection times on Set B. This phase of the work involved the effect of concentration on the Porasil packed columns. A-6 was analyzed at the 1/8 % w/v using 120 and 60 sec injection times. The precision levels for these analyses were calculated.

All of the GPC data were calculated following the usual peak height at the elution mark method with no corrections made for inherent curve errors.

RESULTS AND DISCUSSION

Table IV illustrates the molecular weight data obtained for the narrow distribution polystyrene calibration standards. The Porasil data generally indicated narrower distribution ratios as compared to the Styragel data. Even in the low-molecular-weight range the dispersity values are around 1.1 for the Porasil data. Reliable data is seen on Set B above 20K but overall better agreement with established values was attained by Set A throughout the entire molecular weight range of 2K to 2 million.

Table V illustrates the GPC data from the analysis of our own polystyrene materials. Again the distribution ratios are lower for the Porasil set than those for Styragel. A comparison of the results obtained for Porasil substrate show no significant change in molecular weight as a function of concentration or injection time.

Table VI shows the GPC data resulting from the analysis of the 3 polyvinyl

TABLE VI

GPC DATA	ON	POLY(VINYLCHLORIDE	SAMPLES
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Sample code	Set A-	- Styrag	el	Set B -	– Poras	il						
	1/2% -	— 2 min	2	1/2% -	– 2 mir	e	1/4%	- 2 m	uin 🗌	1/4%	I 1)	iin
<u></u>	M w	\overline{M}_n	D	$\overline{M}_{\iota v}$	\overline{M}_n	D	M _w	\overline{M}_n	D	\overline{M}_w	M _n	D
400-2 (I) (2) (3)	59.75	26.10	2.29	53.40 55.51 58.45	25.62 24.84 25.00	2.23		26.01 24.61		49.65	24.9	1.99
(3) 400-3 (1) (2)	100.25	47.90	2.09	96.93 98.43		1.94				80.70	38.70	2.08
400-4 (I) (2)	152.30 165.52	59.50 64.48		104.53 102.72		2.43	140.5	56.55	2.48	94.1 5	34.10	2.76

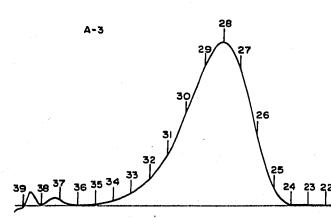


Fig. 3. A-3 polystyrene, 0.5% at $4 \times$ on Set A. 112 k \overline{M}_w , 50 k \overline{M}_n ; 2.22 ratio.

chloride polymers. For the third time Porasil data yields narrower distributions and lower \overline{M}_w and \overline{M}_n values than the Styragel data.

Typical chromatograms of sample: A-3 are shown by Figs. 3 and 4, analyzed on

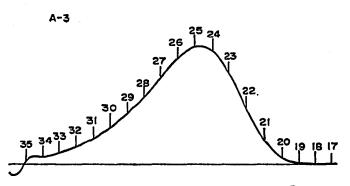


Fig. 4. A-3 polystyrene, 0.5 % at 4 × on Set B. 100 K \overline{M}_{10} , 45 K \overline{M}_{n} ; 2.19 ratio.

TABLE VII

COMPARISON OF GPC DATA (SET A) TO ABSOLUTE VALUES ON PS STANDARDS

Standard	Absolute d	lata		GPC dat	a — Set A		Compari.	sons		
	MI w	\overline{M}_n	MWD	M _w	\overline{M}_n	MWD	ΔM_{iv}	Diff. (%)	$\Delta \overline{M}_n$	Diff. (%)
2.0K	2 0 3 0	1.840	1.10	3 400	3 100	1.10	1 370	67.5	1 260	68.5
5K	5 000	4 600	1.09	4 950	4 5 50	1.09	50	10.0	50	10.8
10.3K	IO 300	9 700	1.06	11 000	9 800	1.12	700	6.8	100	10.3
19.8K	19 800	19 600	1.01	19 950	18 550	1.08	150	0.7	1 050	5.3
51 K	51 000	49 000	1.04	53 650	49 050	1.09	2 650	5.2	50	0.1
98.2K	97 200	96 200	1.01	103 200	92 300	1.12	6 000	6.2	3 900	4.0
160K	173 000	164 000	1.05	157 450	137 700	1.14	15 550	8.9	26 300	16.0
411K	411 000	392 000	1.05	466 400	350 450	1.33	55 400	13.5	4I <u>55</u> 0	10. 6
860K	867 000	773 000	1.12	1 007 500	666 200	1.51	140 500	16.2	106 800	13.8
2145K	2 145 000	1 780 000	1.20	2 165 200	I 134 500	1.91	20 200	0.9	645 450	36 .3

TABLE VIII

COMPARISON OF GPC DATA (SET B) TO ABSOLUTE VALUES ON PS STANDARDS

Standard	Absolute	data		GPC dat	la — Set B		Compar	risons		
	M ,v	\overline{M}_n	MWD	\overline{M}_{w}	\overline{M}_n	MWD	$\Delta \overline{M}_{iv}$	Diff. (%)	ΔM_n	Diff. (%)
2.0K	2 0 3 0	1 840	1.10	7 050	6 600	1.07	5 020	247.2	4 760	258.7
5.0K	5 000	4 600	1.09	8 250	7 750	1.07	3 2 50	65.0	3 1 50	68.5
10.3K	10 300	9 700	1.06	13 400	12 350	1.08	3 100	30.1	2 650	27.3
19.8K	19 800	19 600	1.01	21 100	19 700	1.07	1 300	6.6	100	0.5
51 K	51 000	49 000	1.04	52 100	47 300	1.10	I 100	2.2	I 700	3.5
98.2K	97 200	96 200	1.01	100 950	93 100	1.08	3 750	3.8	3 100	3.2
173K	173 000	164 000	1.05	174 600	157 300	1.11	1 600	0.9	6 700	4.1
411K	411 000	392 000	1.05	436 250	373 650	1.17	25 250	6.1	18 350	4.7
860K	867 000	773 000	J.12	931 950	662 300	1.41	64 950	7.5	110 700	14.3
2145K	2 145 000	τ 780 000	1.20 1	795 400	918 900	1.95	349 600	16.3	861 100	48.8

Set A and Set B, respectively. As seen by the curves, Set A's chromatogram elutes in 12 elution counts while the one from Set B elutes in 16 counts. Visual examination of the two curves leads one to suspect a broader distribution for the Porasil analysis. The calculated data shows almost identical ratios; 2.22 versus 2.23. Since lowmolecular-weight tailing is prominent in Set B's curve and not corrected in the calculations, it seems interesting that the ratios agree so closely.

The GPC data were compared to absolute values established for the PS standards and the resulting data from Set A and Set B are contained in Tables VII and VIII, respectively. One notes the need for correcting the higher-molecular-weight chromatograms due to viscous fingering effects⁷.

Tables IX and X contain the comparisons of GPC data to classical values determined by our laboratory for four out of six samples evaluated by Set A and Set B, respectively. Better agreement is attained by Set A, generally speaking, in particular with \overline{M}_w values determined by light scattering measurements.

TABLE IX

COMPARISON OF GPC DATA (SET A) TO ABSOLUTE VALUES ON BROAD PS SAMPLES

Sample	Absolute val	ues	GPC date	a — Set A		Compar	risons		
	\overline{M}_{w} (light scattering)	M _n (osmometry)	M _w	Mn	MWD		Diff. (%)	∆Mn	Diff. (%)
A-1	33 000	18 200	34 060	17 250	1.97	1 060	3.2	950	5.2
A-3	126 400	51 500	112 880	50 940	2.22	13 520	10.7	500	1.1
A-4	185 500	79 300	150 250	63 050	2.38	35 250	19.0	16 250	20.5
A-5	356 100	165 000	327 380	103 700	3.16	28 720	8.I	61 300	37.1

TABLE X

COMPARISON OF GPC DATA (SET B) TO ABSOLUTE VALUES ON BROAD PS SAMPLES

Sample	Absolute val	ues	GPC dat	a — Set B		Compar	risons	·	
	M _w (light scattering)	M _n (osmometry)	- 171 _w	M _n	MWD		Diff. (%)	ΔM̄ _n	Diff. (%)
A-1	33 000	18 200	31 250	19 230	1.63	1 750	5.3	1 050	5.8
A-3	126 400	51 500	102 530	49 780	2.00	23 870	18.9	1 720	3.3
A-4	185 500	79 300	147 800	73 300	2.02	37 700	20.3	6 000	7.6
A-5	356 100	165 000	278 400	108 830	2.56	77 700	21.8	56 170	34.0

TABLE XI

COMPARISON OF GPC (SET A) TO VENDOR'S GPC DATA

Sample	GPC data	ı (ArRo)		GPC data (Xerox) Set A			Comparisons			
	M _w	Мī "	MWD	M _w	M n	MWD	ΔM_w	Diff. (%)	<i>∆M</i> "	Diff. (%)
400-2	62 350	25 160	2.48	59 75°	26 100	2.29	2 600	4.2	940	3.7
400-3	107 166	45 195	2.37	100 250	47 900	2.09	6916	6.4	2 705	6.0
400-4	117 800	48 850	2.41	158 910	61 990	2.56	41 110	34.9	13 140	26.9

TABLE XII	
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COMPARISON OF GPC DATA (SET B) TO VENDOR'S GPC DATA

Sample	GPC date	a (ArRo)		GPC data (Xcrox) Set B			Comparisons			
	. M 10	М _п	MWD	M w	M n	MWD	⊿M _w	Diff. (%)	ΔM_n	Diff. (%)
400-2	62 350	25 160	2.48	55 780	25 1 50	2.22	6 570	10.5	10	0.03
400-3	107 166	45 195	2.37	97 680	46 525	2.12	9 486	8.9	1 330	2.9
400-4	117 800	48 850	2.41	103 625	44 860	2.32	14 175	12.0	3 990	8.2

TABLE XIII

COMPARISON OF GPC DATA (SET A) TO VENDOR'S ABSOLUTE DATA

Sample	Absolute dat	a	GPC date	a — Set A	- Set A Compari			sons		
	M _w (light scattering)	M _n (membrane osmometry)	MT 10	M _n	MWD	$\Delta \overline{M}_{w}$	Diff. (%)	ΔM_n	Diff. (%)	
400-2	68 600	25 500	59 750	26 100	2.29	8 850	12.9	600	2.4	
400-3	118 000	41 000	100 250	47 900	2.09	17 750	15.0	6 900	16.8	
400-4	132 000	54 000	158 910	61 990	2.56	26 910	20.4	7 990	14.8	

TABLE XIV

COMPARISON OF GPC DATA (SET B) TO VENDOR'S ABSOLUTE DATA

Sample	Absolute dat	a	GPC date	z Set B	et B Compari				
	M _w (light scattering)	M _n (membrane osmometry)	M 10	\overline{M}_n	MWD	ΔMw	Diff. (%)	ΔM_n	<i>Diff.</i> (%)
400-2 400-3	68 600 118 000	25 500 41 000	55 780 97 680	25 150 46 525	2.22 2.12	12 820 20 320	18.7 17.2	350 5 525	I.4 I3.5
400-4	132 000	54 000	103 625	44 860	2.32	28 375	21.5	9 140	16.9

400-4

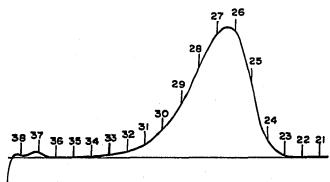


Fig. 5. 400-4 polyvinyl chloride, 0.25% at 16 × on Set A. 158.9 K \overline{M}_{w} , 62 K \overline{M}_{n} ; 2.56 ratio.

In comparing the GPC data from the analysis of the polyvinylchloride polymers, two approaches were taken. Tables XI and XII compared GPC data from Set A and Set B, respectively to the vendor's GPC data. This comparison indicates better

400-4

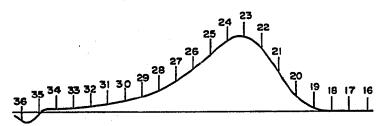


Fig. 6. 400-4 polyvinyl chloride, 0.5% at $4 \times$ on Set B. 103.6 K \overline{M}_{w} , 44.8 K \overline{M}_{n} ; 2.32 ratio.

TABLE XV

COMPARISON OF THE PRECISION BETWEEN THE TWO SUBSTRATES

	X _{Mw}	\overline{X}_{Mn}	X _{MWD}	
Set A A-1 A-6	34.06 ± 0.351 48.37 ± 4.29	17.246 土 0.275 24.39 土 3.286	1.97 ± 0.089 1.98 ± 0.112	·
Set B A-1 A-6	31.25 ± 2.99 43.63 ± 6.82	19.23 ± 5.10 26.93 ± 4.31	1.63 ± 0.26 1.62 ± 0.146	

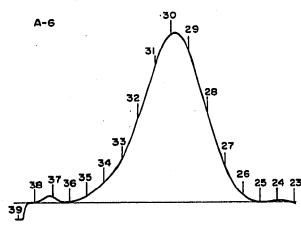


Fig. 7. A-6 polystyrene, 0.5% at $4 \times$ on Set A. 34.06 K \overline{M}_{w} , 17.24 K \overline{M}_{n} ; 1.97 ratio.

agreement is found in Set B especially with \overline{M}_n values, < 10% even at the highest \overline{M}_n value. The highest difference in \overline{M}_w for Set B was 12% as opposed to 34.9% for the same sample on Set A. The second approach was to compare the GPC data to the vendor's absolute data from light scattering and osmometry. The results of this comparison are shown in Tables XIII and XIV. Set A shows better \overline{M}_w agreement than Set B for each sample with somewhat higher discrepancies than seen in Table XI. The opposite effect was observed with the \overline{M}_n values in Set B which had

a

fewer differences than Set A. Figs. 5 and 6 illustrate chromatograms of 400-4 as analyzed on Set A and Set B, respectively.

Two samples, A-1 and A-6, which had been analyzed three times each on Set A and Set B were used to establish precision limits. Table XV contains the results of

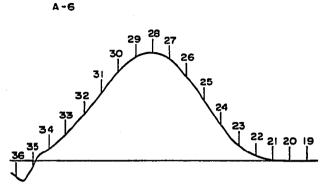


Fig. 8. A-6 polystyrene, 0.5% at $4 \times$ on Set B. 31.25 K \overline{M}_w , 19.23 K \overline{M}_n ; 1.63 ratio.

the precision evaluation. The average precision of Set A was better than Set B. Chromatograms of A-6 analyzed on Set A and Set B are shown in Figs. 7 and 8, respectively.

Reviewing all the data and considering no error corrections made for axial dispersion, diffusion or adsorption, it was difficult to see any great advantage of one substrate over the other. Set A yielded more precise molecular weights in the range evaluated in this study. Set B could be improved by using smaller angstrom packing of higher surface area which was not available at the time this study was initiated. A column packed with the smaller porosity substrate would increase resolution power in the low-molecular-weight region and extend the separation range of the Porasil.

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