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DETERMINATION OF SOLUBLE POLYMER IN ACRYLAMIDE
MONOMER SOLUTION BY AQUEOUS GEL PERMEATION
CHROMATOGRAPHY

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ABSTRACT

An aqueous gel permeation chromatographic method for the determination of soluble polymer in aqueous acrylamide monomer has been developed. Polymer content can be determined in four minutes with consecutive analyses every twenty minutes. The relative precision of the method is better than 2.5% at the 95% confidence level in the range of 100 to 600 ppm polymer. The detection limit for the method is 5 ppm.

INTRODUCTION

The qualitative determination of trace levels of soluble polymer in monomer is normally accomplished by adding a non-solvent to precipitate the polymer and observing the appearance of a haze (1). The quantitative determination of polymer in monomer is determined in a similar manner except that an instrument is used to measure the turbidity (2). The formation and measurement of such precipitates are influenced by a number of variables which usually have an effect on the particle size or the solubility of the polymer. (3,4) The molecular weight distri-

bution of the polymer, the rate of addition of precipitant, the order and time of mixing and impurities can all cause variation in the measured turbidity. As a result, inter-laboratory measurements may give rise to considerably different results on the same sample. It was considered necessary, therefore, to develop a method to measure soluble polymer which was not subject to so many variables.

Gel permeation chromatography (GPC) which separates molecules by their sizes generally has been used to determine molecular weights (MW) and molecular weight distribution (MWD) of polymers. For these purposes, all the molecules have to elute between the total exclusion and penetration volumes of the column. This type of separation can be used to simultaneously determine low MW additives, such as stabilizers and plasticizers, and polymer content in polymeric products in which the polymer is a major component. In the application of GPC for the determination of trace amount of polymer in aqueous solutions of acrylamide monomer, we chose to utilize a column combination such that all the polymer is excluded from the pore volume in order to increase the detection limit, to facilitate quantitation and to eliminate interferences from any low MW components.

EXPERIMENTAL

Instrumentation

A Waters Associates ALC 201/401, including, a M-6000 solvent delivery system and differential refractometer, was used for this analysis. Sample injection was achieved through a Valco high pressure six-port valve with a 0.25 ml sample loop. The detector output was recorded with a Sargent Welch Model (SRG) recorder at a chart speed of 0.1 in./min.

Column and Reagents

The analytical column was stainless steel with dimensions of 50 cm x 4.6 mm I.D. packed with Gly-GPC packing (Electro Nucleonics). The pore size and particle size were 170Å and 37-44 μ respectively. Two such columns were used in tandem. The eluant was 0.1 N reagent grade NH_4NO_3 , adjusted to pH 8.5 with 1 N NaOH. The flow rate was 2 ml/min. Dow polyacrylamide resin #164 was used as a polymer standard.

Procedure

Standard solutions of resin #164 (MW ~400,000) were prepared in deionized water at concentrations ranging from 75 to 600 ppm. Peak height measurements from the GPC runs on these standards were then plotted against concentration to construct a calibration curve which is linear. All subsequent measurements were made in reference to this curve which was found to be reproducible over a period of at least one month.

Samples of 50% aqueous acrylamide monomer solution were introduced directly into the injection loop through a 5 μ Teflon filter.

RESULTS AND DISCUSSION

Figure 1 shows a typical GPC analysis of 50% acrylamide monomer for polymer. The polymer elutes in approximately four minutes and repeated analysis can be achieved every 20 minutes.

Table I demonstrates the precision data achievable with the GPC system. Sample A was a 50% acrylamide solution containing approximately 100 ppm of polymer. Sample B was generated from Sample A by adding resin #164 to make a polymer concentration of 668 ppm. The precision was run over two or more days to show day to day variations.

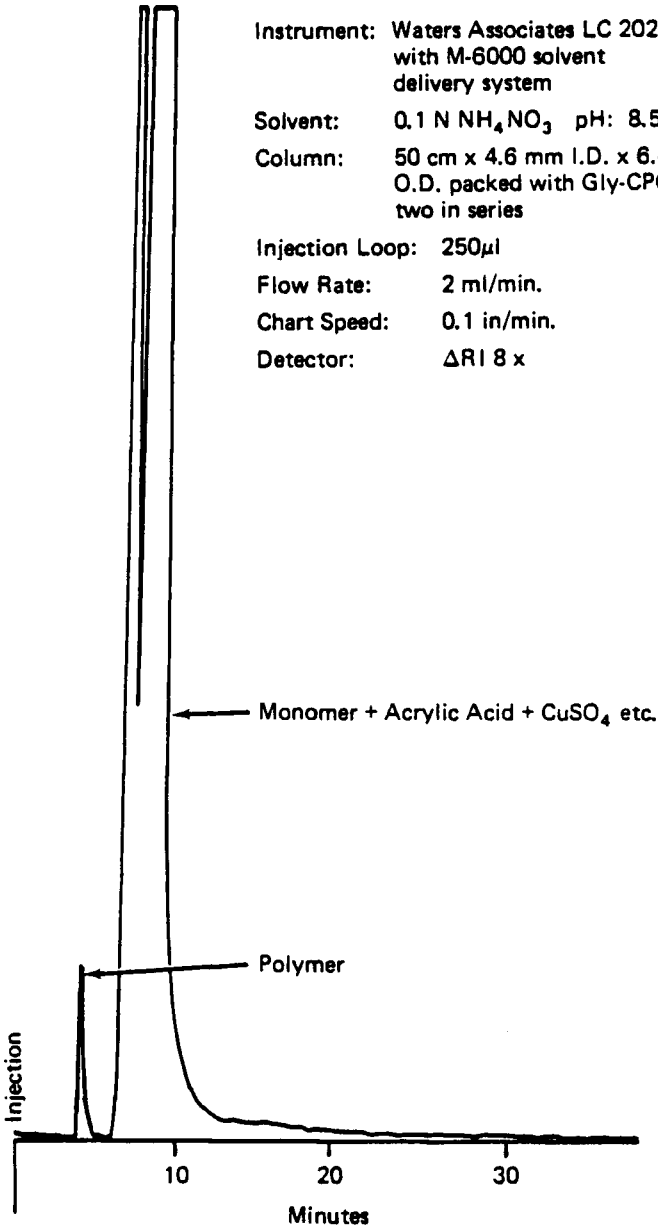


Figure 1

TYPICAL AQUEOUS GPC RUN OF POLYACRYLAMIDE
IN DOW ACRYLAMIDE MONOMER SOLUTION

TABLE I

Precision Measurement for Polymer
in 50% Acrylamide Solution

	Sample A	Sample B (after correction for A)
1st day	104	655
	105	655
	104	660
	104	665
	<u>103</u>	<u>680</u>
2nd day	103	663
	104	658
	103	668
	104	663
	<u>105</u>	<u>665</u>
3rd day	105	
	105	
	105	
	105	
	105	
Average \bar{x}	104	663
Standard deviation σ	1	7.1
Relative standard deviation (95% confidence level)	2%	2.1%

Table II shows recovery data from various amounts of Resin #164 added to 50% acrylamide monomer which originally contained approximately 100 ppm polymer. The detection limit for polymer in 50% acrylamide monomer is 5 ppm under these conditions.

TABLE II
Polymer Recovery from 50% Acrylamide Solution

Concentration Added (PPM)	Concentration Recovered (PPM)	% Recovery
668	655	98
668	655	98
668	660	98.8
668	665	99.6
668	680	101.8
668	663	99.3
668	658	98.5
668	668	100.0
668	663	99.3
668	665	99.6
334	320	95.8
334	330	98.8
167	170	101.8
167	170	101.8
	Average	99.4
	Standard deviation	1.7
	Relative standard deviation (95% confidence level)	3.4%

The molecular weight of soluble polymer in acrylamide is generally observed to be reasonably high, since in all samples analyzed the polymer appears to be essentially excluded from the column packing. To demonstrate that some low molecular weight polymer was not going undetected, a set of standards of equal concentration in water was prepared in the molecular weight range from < 50,000 to 1,000,000. The average molecular weight of these polymers was estimated from viscosity measurements.

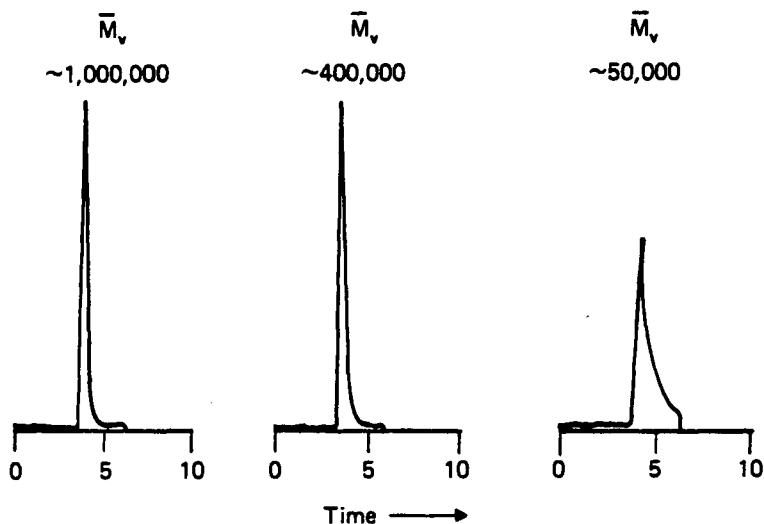


Figure 2

POLYACRYLAMIDE OF VARIOUS MOLECULAR WEIGHTS
AS STANDARD FOR AQUEOUS G.P.C.

Figure 2 shows that the peak height remains constant until low molecular weight polymer is present at which time the peak begins to show a "tail". If this situation arises during analysis, it would be more appropriate to use peak areas rather than peak heights for quantitation.

The GPC analysis for soluble polymer in acrylamide is more specific, more sensitive and less time consuming than turbidimetric procedures. Also, monomer concentrations from 0-50% do not affect the GPC results.

Safety

Acrylamide is a toxic material and the authors strongly recommend the use of goggles and rubber gloves when handling this monomer. The use of a fume hood is also recommended as a further precaution.

Any area of the skin which comes in contact with the monomer should immediately be flushed with copious amounts of water.

Clothing which comes in contact with this monomer should be removed immediately and laundered before reusing.

REFERENCES

- (1) Kline, Gordon M., *Anal. Chem. of Polymers*, XII, 9, Interscience Publishers, Inc., New York, NY.
- (2) ASTM Method D2121-74 p. 29 of 1976 Annual Book of ASTM Standards.
- (3) Klenin, V. J. and Schyogolev, S. Yu., *J. Polymer Sci., Symposium*, 42, 965-972, 1973.
- (4) Klenin, V. J. and Schyogolev, S. Yu., *J. Polymer Sci.*, 44, 181-194 (1974).