



Polyethyleneimine Analysis with HPLC/FID

Application Note

(U)HPLC: Polymers

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Abstract

Polyethyleneimine (PEI) with an average molecular weight of 800 g/mol by light scattering was analyzed from 10 to 1000 ppm with a flame ionization detector (FID) for liquid chromatography (LC/HPLC). A linear carbon-dependent response was obtained.

Introduction

Polyethyleneimine (PEI) is used in several areas from biology and CO₂ capture, to chemicals and electronics. The analysis of PEI for purity and molecular weight distribution is often done using HPLC with light scattering, refractive index, and mass spectrometry detection. Here, we analyze PEI from ca. 10 ppm to 1000 ppm using a novel FID-HPLC detector. In this setup, the solvent is removed from the PEI and the solvent-free PEI molecules are subsequently burned and detected by a catalytic FID.

Experimental

Samples of branched PEI (408719, Sigma Aldrich, average MW ~800 g/mol) were dissolved in HPLC grade water and diluted gravimetrically.

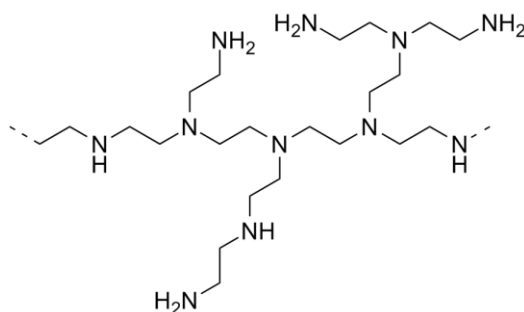


Figure 1. Diagram of a typical branched polyethyleneimine fragment

The samples were injected using an [Agilent 1290 Infinity II LC System](#) without a column, which was connected to the [Solvere](#) (v1, ARC) with a 300 mm x 0.12 mm ID 316SS tube and the following configuration:

HPLC conditions

Column	N/A	
Column temp.	N/A	
Test Standard	Various	
Injection volume	20 µL	
Pump flow rate	0.3 mL/min	
Solvent A	Water, 0.1% formic acid	
Solvent B	Acetonitrile	
Gradient profile:	Time (min)	Percent B
	0	10
	10	10

Solvere™ conditions

Cell Temperature	120 °C
FID Temperature	400 °C
H ₂	50 sccm
Air	350 sccm
Makeup	500 sccm (air)
Pressure	5 psig
Acquisition rate	3.125 Hz
Power	80%
Catalyst	S1-M

Results and Discussion

Figure 2 shows the detector response as a function of time for 10, 50, 100, 500 and 1000 ppm PEI. At 500 ppm the peak width (FWHM) is 0.14 min. with a USP tailing factor of 1.29.

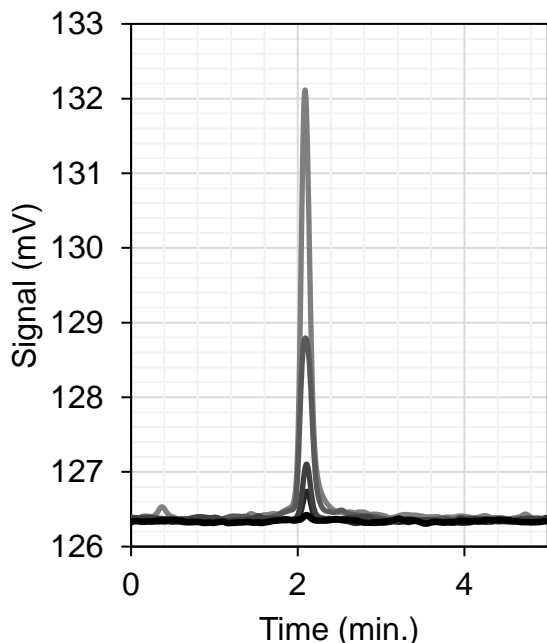


Figure 2. Overlay of chromatograms of polyethyleneimine at 10, 50, 100, 500 and 1000 ppm (dark to light, respectively).

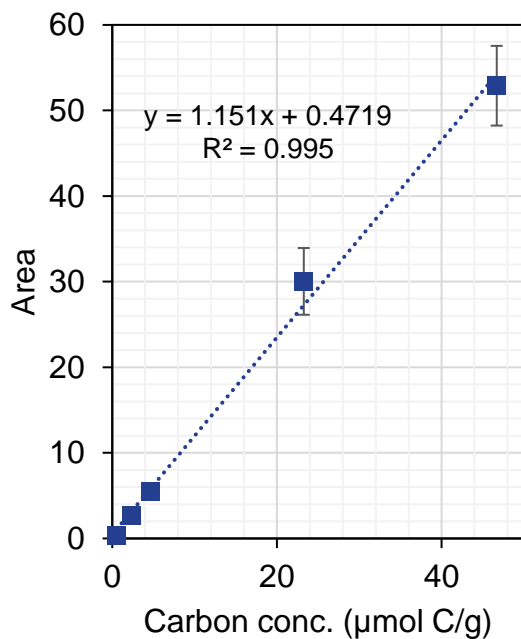


Figure 3. Integrated FID response of polyethyleneimine per carbon from 10-1000 ppm.

The integrated responses of PEI are shown in Figure 3 from 10 to 1000 ppm. The response is linear with a Pearson's correlation coefficient of 0.995. Standard deviations (shown as error bars) of triplicate injections varied from 0.7% to 13% of values depending on the concentration. A minimum detection limit (MDL) of 24 ppm was calculated based on the signal required to give a signal-to-noise ratio of 3.

Conclusions

- Solvere™ CSD gives a linear carbon-dependent response for polyethyleneimine
- Response is highly linear ($R^2 = 0.995$) from 10-1000 ppm
- Minimum detection limit is 24 ppm in the current configuration. Note: this will likely increase with column broadening, e.g., with SEC.

Contact Us

For more information or to purchase a Solvere™, please contact ARC at 612-787-2721, or by email at contact@activatedresearch.com.

Please visit ARC's [website](#) for details and [additional technical literature](#).

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