

FIPA® of Nylon-6,6 in Formic Acid

Introduction:

Nylon is typically analysed in QC by viscosity measurements utilising formic acid or concentrated sulphuric acid as a solvent. This is a lengthy procedure requiring drying, weighing, dissolution, and viscosity measurements. This report describes a simple flow injection polymer analysis (FIPA) procedure for measuring both intrinsic viscosity and molecular weight in formic acid. This is achieved with the use of Viscotek's proprietary I-Series columns and the TDA-302 Triple Detector Array. The I-Series columns are excellent for this application because of their inertness and resistance to strongly acidic solvents.

Experimental:

Dry chip samples of Nylon-6,6 were dissolved in 96% formic acid with 0.02M Sodium Acetate at concentrations in the 2 to 3 mg/ml range. These samples were then filtered with Teflon 0.2µm filters and analysed under the following conditions.

FIPA Run Conditions

Detector: Model 302 TDA (LS-RI-Viscometer)

Solvent: 96% Formic Acid with 0.02M Sodium Acetate

Column: I-OLIGO-3078

Temperature: 50°C

Concentration: 2-3 mg/ml

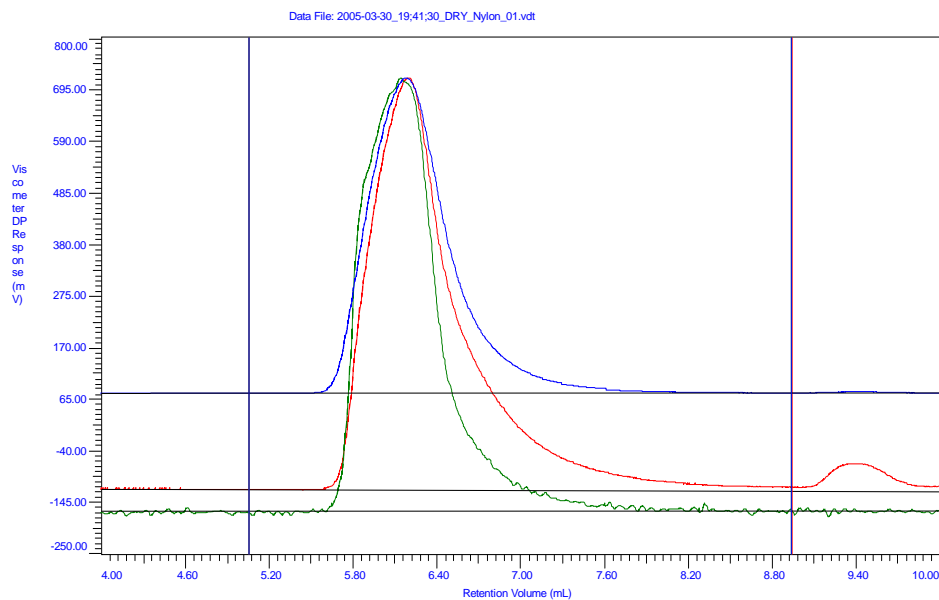
Injection Volume: 100 µL

Flow Rate: 1.0 mL/min

Software: OmniSEC (FIPA method)

Results:

Figure 1 is a typical FIPA-gram representative of the four Nylon samples analysed. FIPA peaks do not separate the polymer according to size like GPC. Instead, the pore size is selected so that the solvent/oligomer peak is separated from the polymer.



The average values of concentration, Mw, and intrinsic viscosity (IV) are obtained by integrating the polymer peaks:

- The refractive index detector peak yields sample concentration, and hence % polymer,
- The light scattering peak yields Mw, and
- The viscometer peak yields intrinsic viscosity.

The following Table I shows the results on the four samples. Three injections are shown to illustrate the precision obtained by the detectors. Of course, precision of the sample analysis itself will also depend on the sample homogeneity, which was not addressed in this study.

Sample Id	Mw	IV	% Polymer
NYLON 0	18,972	0.9357	99.83
NYLON 0	19,164	0.9339	99.73
NYLON 0	19,033	0.9346	99.87
Average	19,056	0.9347	99.81
SD	98	0.0009	0.07
RSD	0.51%	0.10%	0.07%
NYLON 1	17,301	0.8306	99.65
NYLON 1	17,390	0.8298	99.79
NYLON 1	17,317	0.8270	99.90
Average	17,336	0.8291	99.78
SD	47	0.0019	0.13
RSD	0.27%	0.23%	0.13%
NYLON 2	16,456	0.7321	99.77
NYLON 2	16,300	0.7331	99.83
NYLON 2	16,438	0.7327	99.94
Average	16,398	0.7326	99.85
SD	85	0.0005	0.09
RSD	0.52%	0.07%	0.09%
NYLON 3	14,514	0.6530	99.84
NYLON 3	14,527	0.6513	99.74
NYLON 3	14,392	0.6495	100.14
Average	14,478	0.6513	99.91
SD	74	0.0018	0.20
RSD	0.51%	0.27%	0.20%

Conclusions:

In this case, the polymer samples were pure so the % polymer should be 100% if there is no adsorption of sample on the column. This appears to be the case for the I-Series column. The quantitative recovery of sample is important because it opens the door to analyzing impure samples where concentration is unknown.

In conclusion, FIPA is a simple and precise analytical tool for simultaneous measurements of Mw, IV, and % polymer values. The results obtained herein should also apply to Nylon-6 as well as other nylons that dissolve in formic acid.

Key Words: FIPA, Nylon, Polyamide, Formic Acid, GPC, Triple Detection, Mw, IV, % polymer