Viscotek
New Technology in Viscosities and Light Scattering

RELATIVE VISCOSITIES OF 1% POLYAMIDE (NYLON)

Solution Viscosity Application Note 3

Detectors
Viscotek Model 900 Relative Viscometer
SASP Preparation System

The Viscotek 900 Relative Viscometer and SASP preparation system provide excellent results for the solution viscosity of polyamide resins.

In the analysis of Nylon resins, there are three main solvent systems incorporated in the viscosity analysis. 1) Meta cresol 2) 96% Sulfuric acid 3) 90%Formic acid.

The concentration of Nylon samples for Relative viscosity analysis are typically set at 8.4%wt./wt. in formic acid. The 8.4% wt./wt. concentration was set because it represented the limit of solubility of nylon in formic acid. It is also common to find laboratories preparing concentrations of 1% wt./Vol. for the relative viscosity analysis in formic acid or sulfuric acid. The 1% formic acid method provides the quickest analysis as the lower concentration enables quicker and more assured dissolution. Typically, 45 minutes is enough to dissolve a standard molecular weight range Nylon.

Regardless of concentration, the key factors to getting precise data on the RV of Nylons, is borne out in preparing exact concentrations and getting direct measurement of the RV value.

Sample Preparation:

The Semi Automated Sample Preparation (SASP) system developed by Viscotek uses a balance and syringe pump coupled by a PC computer. It is based on the principle that it is easier to adjust the volume of a liquid than the mass of a solid. The operator weighs a sample into a target range. The computer takes in the weight from the balance via an RS232 connection. The amount of solvent dispensed by the syringe pump is calculated using concentration, density for wt./wt. measurements, % sample impurities, and the expansion of the solvent from preparation temperature to analytical temperature for wt./vol. measurements. By carefully accounting for common errors in preparation, the system typically provides far greater precision in preparation and a safer method to handle solvents.

Vial	Sample (g)	Volume (ml)	Room Temp Conc. (g/dl)	Analytical Temp Conc. (g/dl)
1	0.2722	27.20	1.00074	1.00041
2	0.3128	31.28	1.00016	.99983
3	0.2961	29.60	1.00034	1.00001
4	0.3072	30.70	1.00065	1.00032
5	0.2888	28.88	1.00017	.99984
6	0.2880	28.80	1.00000	.99967
7	0.3157	31.55	1.00064	1.00030
8	0.2717	27.15	1.00074	1.00040
9	0.3135	31.35	1.00000	.99967
10	0.2649	26.48	1.00057	1.00023

Data for 1% wt./vol. preparations:

In preparation of the samples it is important to consider the following sources of error.

- Errors in the concentration present a logarithmic change in the RV of the solution. A 0.1 increase of concentration is approx. an increase of 1.5 RV units at an 8.4% RV of 50
- Moisture in the Nylon creates error in nylon mass. A 4% moisture content will cause an approx. 1% lower RV.
- Degradation of a nylon solution in formic acid occurs at a rate of about 5% per 24 hours.
- Deviation of the concentration of formic acid from 90%. An increase of 0.1% yields an approx. increase of 0.2% RV.
- A temperature increase of 0.1 degree will cause a lower viscosity value of approximately 0.1 RV units.

Sample Analysis

Due to the High RV's experienced in the traditional nylon tests; it is difficult to make a direct comparison of sample viscosity to solvent viscosity using the traditional glass capillary tubes. It therefore becomes necessary to calibrate different tubes, using different calibration oils, and make a ratio of the viscosities in separate measurements. This method leaves the test prone too much experimental error. However by employing a dual capillary relative viscometer the capillaries can be adjusted for the viscosity range and the comparison of solvent to sample can be made directly, easily, and precisely.

Sample	Relative Viscosity
1	2.403
2	2.401
3	2.403
4	2.398
5	2.395
6	2.409
7	2.405
8	2.396
9	2.398
10	2.401
Avg.	2.401
Std.Dev	0.0043

Data for the analysis of Nylon 6,6 at 1% wt./vol.

In addition to the precision of the data demonstrated above, the relative viscometer, presents a easy opportunity to automate a particularly laborious task freeing up operators to perform other vital laboratory functions. Automation can range from simple autosampler systems to full robotics ability to serially prepare and run samples unattended.

15600 West Hardy Road · Houston, Texas 77060, USA · 713/445-5966 Fax: 713/931-4336