

Experience the new Model 575 Intrinsic Viscometer Generation 2

The Most Rapid and Reliable Measurement of Molecular Weight and Intrinsic Viscosity





In a three-way comparison of the best market alternatives, a major polymer manufacturer determined that **Haiku**:





Applications: Intrinsic Viscosity

Polyesters and PET



Polycarbonate



Polyvinyl Chloride



Polystyrene



Biopolymers



Polyolefins



Viscometers for Plastics and Polymers Designed and Manufactured in Houston, Texas 713-724-2890 | admin@haikuinstruments.com www.linkedin.com/company/haiku-instruments

<u>What is Intrinsic Viscosity, [ŋ]?</u>

Answers abound:

"the limiting value of the ration of specific viscosity to concentration as the concentration approaches zero"

"a measure of the polymer's contribution to the viscosity of a solution in the absence of intermolecular interactions"

"the *inverse density* of the polymer, indicating the fluid volume it excludes from the solution per unit weight"

"a rapid and precise way to measure bulk MW"

The practical application of Intrinsic Viscosity measurement is the calculation of Molecular Weight.

Represented by the Greek letter eta **[ŋ]** and bracketed [] to indicate its distinction from other different viscosity calculations, intrinsic viscosity relates to molecular weight by the formula:

[η] = K * M^α

Where

K and α are constants specific to the polymer-solvent system and M is the viscosity average molecular weight of the polymer.

In short: with a known Intrinsic Viscosity, one can directly calculate a polymer's average molecular weight.

Intrinsic Viscosity– where to begin?

Intrinsic Viscosity is derived from Relative Viscosity by way of Specific Viscosity, in the sequence shown here:

 $\eta_{relative} = \eta_{sample} / \eta_{solvent}$

$$\eta_{specific} = \eta_{relative} - 1$$

 $[\boldsymbol{\eta}] = \lim_{c \to 0} \frac{\eta_{specific}}{c}$

Because so few variables are involved, these three parameters are critical for Intrinsic Viscosity:

relative viscosity | concentration | temperature

Accurate measurement of relative viscosity, tight temperature control, and accurately prepared samples are the main factors contributing to results.

How do we measure Relative Viscosity (RV)?

The Modern Method The Historical Method time-based, gravity flow pressure-based, forced flow Ubbelohde Tube Viscometer sample loop -0007Set Volume capillary 1 capillary 2 injection pump Capillary valve waste Reservoir syringe pump

Foundational work in polymer science was carried out using tube viscometers of the Ubbelohde and Ostwald types.

Polymers pioneers spent countless hours making exacting measurements using these glass-tube devices.

Today, automated glass tube viscometers use electric-eye sensors and vacuum-based sample loading with air and solvent cleansing of the glass tubes.

However...

Best-in-class glass tube viscometers are in principle unable to compete with forced-flow viscometers!

The Dual Differential Viscosity Measurement



The beginning of each run includes a solvent **baseline**,

followed by an **injection** of the dilute polymer sample,

ending with a **plateau** at the new equilibrium.

From the ratio of the plateau to the baseline: relative viscosity.

Each measurement includes both solvent and sample viscosity.

From **Poiseuille's Law** we know that the difference in pressure across a tube of length L and radius R at flow rate Q is as follows:

$$\Delta P = \frac{8\eta LQ}{\pi R^4}$$

Solving for viscosity, we rearrange this to:

 $\eta = \frac{8LQ\Delta P}{\pi R^4}$

Considering the relative viscosity of the dilute polymer sample to the solvent, we can divide these and obtain the following equation:

Relative Viscosity (RV) = $\frac{\eta_{sample}}{\eta_{solvent}} = \frac{\Delta P_{sample}}{\Delta P_{solvent}}$

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Sample Preparation + Dissolution + Analysis

The completely integrated solution IV system... with one minute user time per sample!

Preparation

Input sample identifier, name, or lot number Press "Begin Sample Weighing" Follow the Guided Weighing Workflow, sample by sample Linked analytical balance tares vials, records sample masses Add miniature stir bar to the vial and twist on septum-cap Place sample vials into heated autosampler dissolution block Sample Mass / Concentration Target = Solvent Volume Syringe Pump delivers solvent to each sample automatically

Dissolution

Individual Digital Stirrers engage at each sample location Stirring Program is Based on Solvent Viscosity and Sample IV Dissolution Timer Starts After Dispensing

<u>Analysis</u>

System Suitability Check ensures ratio stability and linearity Sample 1 is loaded, injected and analyzed. Twice. All subsequent samples follow this analysis scheme Sequence End method purges and flushes the system with solvent Remove spent vials from the autosampler dissolution block

Only the Steps in **Blue** Require User Attention!

Sample Considerations and Concerns

Expected IV (dL/g)	Concentration Target (g/dL)	Sample Mass Target ± 15% (mg)
0.03 to 0.1	6	1200
0.1 to 0.3	2	400
0.3 to 0.5	1	200
0.5 to 0.7	0.8	160
0.7 to 1.5	0.4	80
1.5 to 3	0.2	40
3 to 10	0.1	20
10 to 20	0.05	10
20 to 30	0.02	4
30 to 40	0.01	2

Concentration Targets and Sample Mass Requirements

k-Value and Intrinsic Viscosity for PVC

k-Value is a calculation using RV and sample concentration used more often than Intrinsic Viscosity in PVC markets outside North America. A direct side-by-side calculation of both the *k*-value and IV is available for PVC users.

Filled and Finished Samples

Glass fibers, carbon black, and inorganic fillers reduce the polymer fraction of the sample. A calculation of the % polymer fraction of the sample mass can be enabled either before or after sample analysis, fitting your analytical lab workflow.

Inline Filtration and Automatic Backflush

When samples contain filler materials or gel not suitable for dilute solution analysis, glass tubes plug and fail. No so with our capillary viscometers! Insoluble particles of all types are filtered out during sample loading. Then they are backflushed to waste as needed per application, as well as after all samples have been analyzed.

Integrated Sample Weighing Protocol with a Connected Balance

Currently prepping sample name: Polyamide A Clear off balance for zeroing Zero balance Place empty vial on balance Tare balance Weigh out the sample Add stir bar/cap to vial and place in the autosampler			Current	: Weight: 0.0000 g	Target Weight: 0.04	20 g Min Weight: (0.0380 g Max Weig	ht: 0.0460 g	
	Sample Name	Position	Status	Conc. (g/dL)	Calc. Polymer Weight (g)	Act. Conc. (g/dL)	Calc. Volume (mL)	Dissolution Start Time	Prep Comp. Time
1	Polyamide A	1	PreWeigh	0.200	0.0400	0.200	20.000	-	-
2	Polyamide B	2	PreWeigh	0.200	0.0413	0.200	20.650	-	-
3	Polyamide C	3	PreWeigh	0.200	0.0410	0.200	20.500	-	-
4	Polyamide D	4	PreWeigh	0.200	0.0405	0.200	20.250	-	-

LIMS / ERP Integration and/or CSV Export Options are Standard



Data Overlay for Visual Comparison of Material Grades



FAQ's about Haiku Instruments and Solution Viscometry

Q: We have NEVER heard of Haiku Instruments. Where did your company come from? A: Our design and engineering team grew from experience building, repairing and servicing the Viscotek / Malvern Instruments Y501 platform which dominated the solution viscosity market in the 1990's-2010's.

Q: What makes Haiku Instruments the right choice for Intrinsic Viscosity or k-value?

A: Based on the ASTM D5225 standard and implementing our patented (2023) improvements, users can – with only one minute operator time – analyze (at minimum) triple the sample workload versus any other viscometer.

Q: Does flow rate fluctuation from a solvent pump impact the measurement?

A: With a single capillary, it certainly would! 1970's-1980's viscometers did this. In our configuration, two ΔP measurements are made at all timepoints: $\Delta P1$ always measures the solvent, while $\Delta P2$ measures the solvent at **baseline** and the sample at **plateau**. Any variability in flow rate (Q) and subsequent fluctuations in the pressure (ΔP) cancel out.

Q: Why does your automated system cost around \$100,000 when I can buy a glass tube and a stopwatch, measuring IV with less than a \$500 investment?

A: Because your company cares about your workload, your safety, the accuracy of the results and the efficiency of the laboratory operations. Manual sample preparation and analysis expose users to solvent vapors at each step, while simultaneously decreasing measurement accuracy. Users spend upwards of 60 minutes for a single sample in this way, versus a single minute of operator time for our automated system.

Q: How much time and effort, solvent and consumables will I save by upgrading?

A: 3x the sample throughput is usual. 3-20x the user time, depending on your current system. 3-5x solvent savings on use and disposal costs. One vial/cap per sample is all we require. No further consumables are needed for daily operation.

Q: Can we send in some polymer samples for trial analysis?

A: Absolutely! With an SDS and arrangement, we will gladly run most polymer samples.

Q: How do I have confidence in the results or verify measurement accuracy? A: The final test of system suitability in a dual-capillary viscometer is *linearity*. An increasing flow rate through capillary R1 and R2 should result in no significant change in the ratio of the pressure signals. This means that the requirements surrounding Poiseuille's law regarding flow rate, temperature, tubing length and diameter are all met. Any RV measurement is then based on the viscosity of the solutions, and no other external factors.

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Specifications Overview

Model 575 Intrinsic Viscometer, Gen 2

Workflow Step	Critical Advantages				
Sample Mass Determination	Guided Weighing with connected Balance				
Solvent Dispensing	Fully Automated; zero user solvent handling				
Dissolution	24 Individually Stirred Autosampler Positions				
Sample Analysis	Begins automatically after dissolution				
Criterion	Specification				
Viscosity Measurement Type	Dual Differential, Relative Viscosity, Forced Flow				
IV Measurement Resolution	0.005 dL/g				
Measurement Precision	Better than 0.2% RSD RV @ 0.800 dL/g				
Shear rates	200-500 s ⁻¹ (typical, depends on application)				
Sample Analysis Time	4-6 minutes per sample, including duplicate injection				
Solvent Compatibility	Organic, Aqueous, Acids, Halogenated				
Temperature Range (Dissolution)	30°C to 160°C				
Temperature Range (Analysis)	4°C to 160°C				
Solvent Per Sample (prep + analysis + wash)	25mL				
Data and Results Integration Options	LIMS/ERP / Parsing / Excel / CSV				
Compliance Support	21CFR part11				
Connection and PC Operating System	USB / Windows 10/11				

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