Viton® FreeFlow™
From DuPont Performance Elastomers

How to Measure Processing Additive Concentration

For most applications, the amount of processing additive contained in the resin is a critical factor in performance. Measuring the additive at these low levels (50–1500 ppm) can be difficult. The discussion below will detail available methods for measuring the fluoropolymer level. However, having the proper level of a fluoropolymer present does not guarantee the desired benefits.

The performance of any resin containing a fluoropolymer additive depends on many factors. Those factors under control of the resin or masterbatch producer are the type of fluoropolymer, the additive level, the dispersion quality, and interaction effects. Other factors, outside the control of the producer, are other ingredients added by the processor, die surface contamination, and operating conditions.

When considering the use of a processing additive, it is important to choose the fluoropolymer type wisely. Viton® FreeFlow™ 10 can be described as the original type of fluoropolymer processing additive. More recently, the fluoropolymer alloys Viton® FreeFlow™ SC or Viton® FreeFlow™ RC offer increased efficiency and reliability that makes them more cost effective for most applications. Its low cost of use also makes it practical for many situations where additives may not have been considered in the past.

Dispersion quality can best be evaluated through some form of microscopy. This can be tedious when the fluoropolymer is present in the resin at a level of 250 ppm. Unfortunately, the presence of antiblock or pigment makes evaluating the dispersion even more difficult.

Formulation interactions are also difficult to predict. When the additive interacts with other additives the efficiency of the additive is reduced. These interactions are a function of both the relative levels of the various additives and the amount of mixing that the resin experiences.

The best way to determine if an additive is both present and functioning is to use some form of extrusion testing. The resin should be extruded through a clean extruder and die. Die pressure and extrudate appearance are monitored over time and compared to established standards. This type of test measures how well the additive is functioning. It incorporates level, dispersion, and interactions in one test. Any extrusion testing requires establishing careful controls and following rigid equipment cleaning procedures.

Measuring Additive Level

In a typical formulation the additive level may vary greatly from one pellet to the next. A resin containing processing additive masterbatch may have one pellet containing 2% (20,000 ppm) fluoropolymer for every 50 pellets with none. A fully blended resin averaging 200 ppm of fluoropolymer may have pellets ranging from 0–400 ppm. Although this variation will not adversely affect performance, it does complicate accurately measuring the level.

For these reasons it is important to determine the average level across a fairly large sample. To get a representative value, a large sample should be homogenized using a Banbury or other intensive mixer. This will change the dispersion and increase the interactions but not change the fluoropolymer level.

The methods available for determining level are summarized in *Table 1*. Some measure the fluorine directly and some use an indirect method for determining the amount of fluoropolymer present. The best choice depends on the application and the available equipment.

Table 1
Analytical Methods

Neutron Activation Energy	
Technique	Elemental analysis
Sensitivity	1 ppm or less
Advantages	Very accurate
Disadvantages	Expensive equipment, slow process
Comments	Testing done by universities or large research lab, not practical for process control
Combustion Analysis	
Technique	Convert fluoropolymer to HF, dissolve in water, and measure fluoride ion concentration
Methods	Wickbold torch, Parr bomb, Schöniger flask, specific elemental analyzer
Sensitivity	1–10 ppm
Advantages	Accurate, small sample needed
Disadvantages	Can be slow, only uses small sample, can require wet chemistry
Comments	These methods have been used for some time with a great deal of success. The recently introduced elemental analyzers for fluorine can give results in 15 min. There are contract laboratories that can have results overnight.
X-Ray Fluorescence (XRF)	
Technique	Compares x-ray emissions to standards
Sensitivity	10%
Advantages	Rapid
Disadvantages	Needs extensive controls; direct measurement of fluorine requires special XRF equipment
Comments	This method requires uniform thickness film samples and carefully developed standards.
Infrared Analysis	
Technique	Passes infrared light through material; amount of light absorbed in specific wavelengths is compared to identify relative amount of different compounds present
Methods	Fourier Transform Infrared (FTIR), Near Infrared (NIR)
Sensitivity	50 ppm
Advantges	Rapid; easy, inexpensive equipment
Disadvantages	Needs accurate controls, silica and other additives can interfere with anlaysis
Comments	These methods are accurate down to the 1% level. They can show the presence of a fluoropolymer at the 200 ppm level. Useful for concentrates but not very useful for fully compounded resins. Online FTIR analyzers are available.
Thermogravimetic Analysis (TGA)	
Technique	Compares weight loss at various temperatures
Sensitivity	2%
Advantages	Rapid; can check several components
Disadvantages	Small sample size, only for high (>1%) concentrations

Density	
Technique	Compare density of sample with known standards
Sensitivity	1,000 ppm
Advantages	Rapid; standard test using density column
Disadvantages	Other additives affect results; needs void-free sample; only works at high levels
Comments	Needs good controls; only good for high concentrations (masterbatches).
Weigh-up	
Technique	Record amount of material as it is added; maintain weigh-feeder records
Sensitivity	Depends on feeders
Advantages	No post-production testing
Disadvantages	Relies on equipment or operator accuracy
Comments	Used extensively in combination with other methods to monitor results.
Performance	
Technique	Extrude resin and measure conditioning rates
Advantages	Measures performance; includes dispersion and interactions
Disadvantages	Off-line extrusion testing, requires cleanup after testing, extensive standards
Comments	Measures what the additives are present to do; effects of interactions and dispersions are included in the testing.

For further information please contact one of the offices below, or visit us at our website at www.dupontelastomers.com/viton

Global Headquarters — Wilmington, DE USA
Tel. +1-800-853-5515
+1-302-792-4000
Fax +1-302-792-4450

European Headquarters — Geneva
Tel. +41-22-717-4000
Fax +41-22-717-4001

South & Central America Headquarters — Brazil
Tel. +55-11-4166-8978
Fax +55-11-4166-8989

Asia Pacific Headquarters — Singapore
Tel. +65-6275-9383
Fax +65-6275-9395

Japan Headquarters — Tokyo
Tel. +81-3-6402-6300
Fax +81-3-6402-6301

(08/98) Printed in U.S.A.
Reorder no: VFE-H79972-00-A0698

DuPont
Performance Elastomers