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FLOW INJECTION POLYMER ANALYSIS (FIPA) OF EPDM ELASTOMERS

Triple Detector Application Note 12

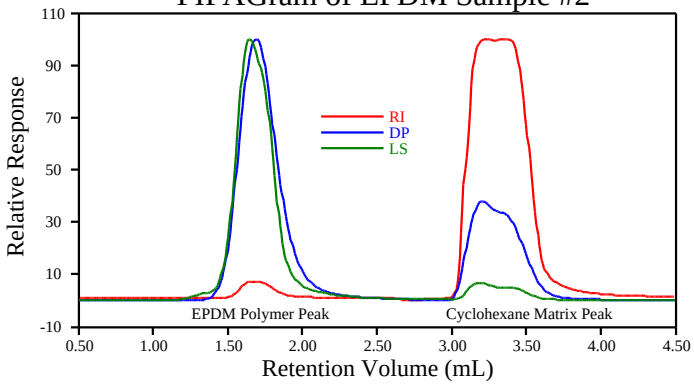
Ethylene-Propylene-Diene-Monomer (EPDM) Elastomers are random amorphous polymers with outstanding properties and broad applications. These materials are commonly found in automotive parts, single-ply roofing membranes, motor oil formulations etc. Historically, Molecular Weight Distribution (MWD) characterization of EPDM has been carried out using High Temperature GPC. Common run conditions involve the use of trichlorobenzene as the solvent and 140°C temperature. The FIPA method described below provides an alternative chromatographic method that is much more user friendly.

FIPA Run Conditions

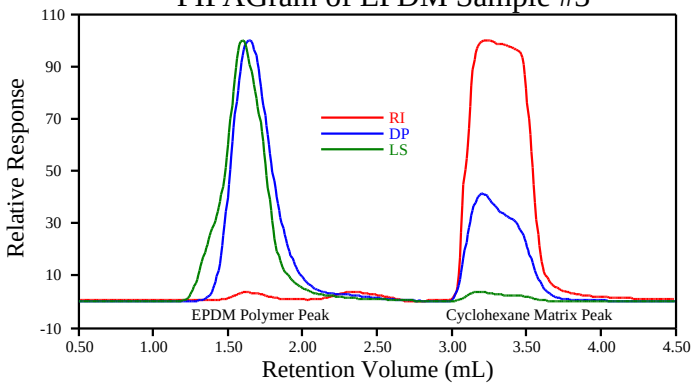
Detector: Model 300 TDA
Solvent:: THF
Column: ViscoGEL FIPA-100H (10cm X 7.8mm id)
Temperature: 60°C
Concentration: Approximately 0.2%
Injection Volume: 100 µL
Flow Rate: 1.0 mL/min

In this application, the EPDM Samples are first dissolved in hot (60 to 80°C) cyclohexane. The time and temperature required for this dissolution can vary depending on MW and crystallinity of the individual samples. Please note that most EPDM samples are not soluble in THF, even up to the boiling point of THF. However, we were able to directly inject samples dissolved cyclohexane into a 60°C THF FIPA system and obtained excellent molecular results. The following is a couple of example EPDM FIPA chromatograms (FIPAGrams).

FIPAGram of EPDM Sample #2



FIPAGram of EPDM Sample #3



Please note that the FIPA-100H (100 angstrom) column is doing a very good job separating the polymer peak from the cyclohexane solvent peak (in about 4 minutes). However, there is little or no resolution of the polymer distribution. Please note the similarity of FIPAGrams of samples #1 and #3 which are quite different in Mw values. Therefore, FIPA can only provide MW and IV averages (i.e., no distribution information).

The following table is a tabulation of FIPA results for a set of high crystallinity EPDM samples.

ID	Mw	SD	%RSD	IVw	SD	%RSD
#1	133,000	1,060	0.80	1.534	0.004	0.28
#2	119,000	430	0.35	1.321	0.002	0.18
#3	1,329,000	1,960	0.15	1.144	0.003	0.26
#4	1,216,000	7,600	0.63	1.544	0.013	0.83
#5	605,000	1,560	0.26	1.364	0.005	0.37
#6	189,00	130	0.07	1.715	0.006	0.35

It is clear here that the repeatability of these FIPA runs (4 injections for each sample) are excellent for both MW and IVw. Next, we compare the FIPA results to GPC under similar conditions.

GPC Run Conditions

Detector: Model 300 TDA
Solvent:: THF
Column: ViscoGEL GMHHRHH (30cm X 7.8mm id)
Temperature: 60°C
Concentration: Approximately 0.2%
Injection Volume: 100 µL
Flow Rate: 1.0 mL/min

The following is tabulation of GPC and FIPA results for the same three samples. All samples were run in triplicates.

ID	Mw	SD	%RSD	IVw	SD	%RSD
FIPA-A	157,000	1,250	0.80	1.805	0.005	0.28
GPC-A	163,000	5,700	3.50	1.874	0.046	2.46
FIPA-B	140,000	500	0.36	1.554	0.002	0.13
GPC-B	148,000	3,200	2.16	1.654	0.031	2.16
FIPA-C	222,000	150	0.07	2.018	0.007	0.32
GPC-C	201,000	5,200	2.57	2.222	0.021	0.94

The averaged RSD for Mw is 0.41% for FIPA and 2.74% for GPC. The averaged RSD for IVw is 0.24% for FIPA and 1.85% for GPC. This represents approximately a 7-fold gain in precision for the FIPA technique. This gain in precision is also accomplished in about 1/3 of the GPC analysis time. In terms of the actual values, we believe that the two techniques provided comparable results. The FIPA data was gather at the Viscotek Houston Laboratory while the GPC data was provided by the Bayer Laboratory at Sarnia, Canada.

In conclusion, FIPA is a fast, precise and accurate analytical tool for routine process and quality control. This "PULL, DILUTE and SHOOT" technique provides quick and easy simultaneous measurements of Mw, IVw, and %Polymer for liquid process samples. For a more thorough basic description of the FIPA technique, please refer to Triple Detector Application Note 11.

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