CRYSTEX: Automated Xylene Solubles in Polypropylene with Viscometer and Composition Detectors

A. Ortín, B. Monrabal, L. Romero. Polymer Char, Valencia, Spain.

Introduction

The established CRYSTEX technique, based on separation of cycle plus filtration inside of a stainless steel vessel, provides very precise results in the determination of the xylene solubles percentage, also known as xylene extractables (XS), in polypropylene. The full automation of CRYSTEX makes it a very convenient alternative to manual (ISO 6427, part B) or other methods used in the industry.

The addition of a capillary viscometer as well as special IR sensor, for ethylene content determination, to the CRYSTEX instrument creates a very powerful tool for the analysis of polypropylene copolymers. A schematic diagram of such an instrument is presented in Figure 1, together with the parameters being measured directly: Whole sample at high temperature after dissolution cycle and amorphous fraction (rubber) at low temperature after crystallization. All information available from a single experiment is summarized in Table 1 for a series of different samples.

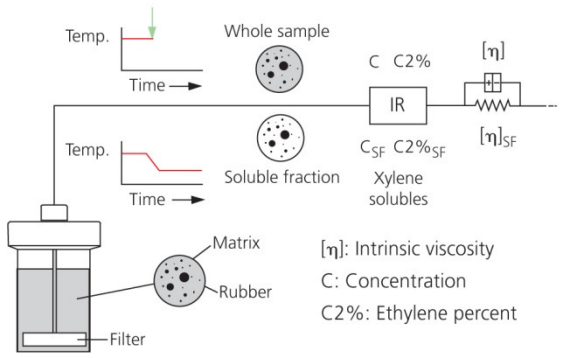
Results

The capillary viscometer implemented into the CRYSTEX makes it possible to obtain the intrinsic viscosity [η] of the original sample as well as of the amorphous and crystalline components. Polymers in a very broad range of viscosity can be measured providing good precision better than 0.7% RSD.

Specific sensors responding to the absorption bands of methyl and methylene groups in the MID-IR are used in order to analyze the initial polymer as well as the amorphous phase providing composition information in terms of ethylene percent (C2%).

Full composition range is covered with the same calibration keeping a very good precision of less than 0.1% of ethylene, in the original sample.

Figure 1: CRYSTEX diagram measured parameters.



Conclusions

A precise and robust apparatus has been made available to fully characterize PP copolymers. The intrinsic viscosity and ethylene contents of the whole polymer and its amorphous and crystalline fractions can be determined, as well as the soluble fraction percentage, which directly relates to xylene solubles, in a single analysis using a fully automated instrument under computer control. Thus a very comprehensive characterization of PP is achieved with a single instrument requiring very little operator interaction.

Table 1: Summary of results obtained in each single CRYSTEX analysis (different samples).

Sample	Whole polymer		Amorphous		XS %	Crystalline (matrix)		
ref.	[η] dL/g	C2%	[η] dL/g	C2%		[η] dL/g	C2%	β/α
A	3.019	8.8%	4.351	31.6%	12.23%	2.834	2.8%	1.54
B	1.096	10.1%	1.494	32.7%	16.12%	1.020	1.0%	1.46
C	1.392	13.4%	1.636	34.4%	17.77%	1.339	1.3%	1.22
D	1.873	10.5%	3.326	34.3%	15.21%	1.612	1.6%	2.06

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