Company profile

Polymer ChAR

Polymer ChAR is a high-technology company devoted to the development of state-of-the-art instrumentation for Polymer Analysis. It provides, as well, analytical, fractionation and consulting services in the field of Polyolefins Characterization.

Polymer ChAR was founded and established in 1992 at the Technology Park in Valencia, Spain, with the initial goal of developing a commercial CRYSTAF instrument. CRYSTAF (Crystallization Analysis Fractionation) is a new technology patented by DOW to measure the Chemical Composition Distribution in semicrystalline polymers.

Polymer ChAR has introduced in recent years a broad range of products for Polymer Analysis and more specifically for Structural Characterization of Polyolefins like a fully automated TREF (Temperature Rising Elution Fractionation), a dedicated CRYSTAF instrument for quality control of multireactor - multicatalyst resins, the CRYSTAF-QC, a bench top instrument for preparative fractionation of polymers by composition or molecular weight, the PREP-mc2 apparatus, an automated instrument for fast Xylene extractables analysis in Polypropylene, the Cryst-EX, and more recently the new optoelectronic Infrared detector, IR4, for concentration and polyolefins composition analysis when attached to a high temperature GPC or as a general use composition detector in HPLC.

Polymer ChAR offers now optional Light Scattering and Viscometry detectors attached to CRYSTAF, TREF or combined CRYSTAF-TREF instruments for full characterization of complex polyolefin resins.

Polymer ChAR is well known by its advanced approach into virtual instrumentation software, that together with excellent Remote Control capabilities and its strong commitment to customer success, places the company in the leading edge on instrumentation Diagnostics and Support Service.

Cryst EX

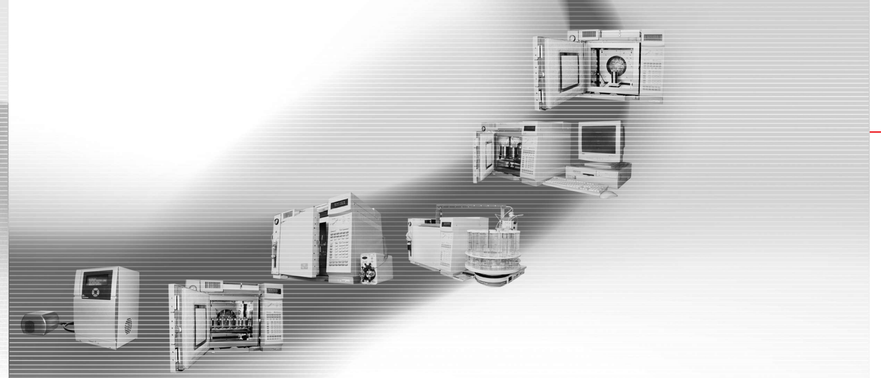
Cryst-EX is a fully automated technique for the fast and precise analysis of the Xylene Extractables / Heptane Solubles in Polypropylene resins with a simple and robust instrument. The overall analysis time is less than 3 hours. The incor-poration of an additional Infrared sensor and a single capillary viscometer makes of Cryst-EX a very powerful apparatus still being adapted to be used in a Quality Control Laboratory.

Capabilities

- Simultaneous analysis of 3 samples in less than 3 hours.
- Up to 1 gram of sample can be analyzed to improve sample homogeneity.
- Incorporation of the new digital IR4 detector for composition analysis of the extractables and the original sample.
- Possibility of incorporation of a simple Viscometer detector.
- Linear correlation with Xylene extractables ISO method.
- Linear correlation with Heptanes soluble.

Advantages

- Fully automated analysis with simple hardware built in a benchtop instrument.
- High precision (better than 5%).
- No need of weighting the sample.
- Fast analysis with little manpower intervention. Samples introduced every 3 hours.
- Lower consumption of solvent and less flammable.
- Designed for Quality Control environment (easy to use and easy to maintain).
- Remote control access for easy diagnosis and software upgrades.



Products from Polymer ChAR

- CRYSTAF model 200 plus: An instrument designed for intensive use in the analysis of the Chemical Composition Distribution in Polyolefins, providing a broad structural data base to improve catalyst know-how and allow new resins design.
- TREF model 200 plus: A completely automated apparatus for the analysis of the Chemical Composition Distribution in Polyolefins by TREF. It provides complementary information to CRYSTAF data in the analysis of some complex resins.
- Crystaf QC: A simple and robust apparatus for the precise and fast analysis of the Chemical Composition Distribution in a Quality Control environment.
- Cryst-EX: A new apparatus specially designed for the analysis of extractables (amorphous-soluble fraction) in polypropylene in a Quality Control environment.
- IR4: A new high sensitivity and reliable IR detector that can work using up to four simultaneous wavelengths to measure concentration and composition.
- PREP mc2: An automatic semipreparative fractionation instrument to perform fractionation of polymers according to Composition or to Molecular Weight.
- Analytical Services: Polymer ChAR provides a world wide polymer characterization and fractionation service.

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Cryst EX

A new approach to the analysis of extractables in Polypropylene

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Polymer ChAR

Leader in Polyolefins Characterization

Cryst EX

A new approach to the analysis of extractables in Polypropylene

A new apparatus has been built to measure the extractables level in Polypropylene. When properly calibrated Cryst-EX provides equivalent numbers to the Xylene Extractables (ISO 6427, App. B). The equipment, built for a Quality Control environment, performs all the operations automatically with no manpower intervention other than introducing the sample in the crystallization vessel. Three differents samples can be analyzed at once in less than 3 hours.

The Heptane solubles according to ISO method can be measured as well with the Cryst-EX apparatus.

Analytical Process

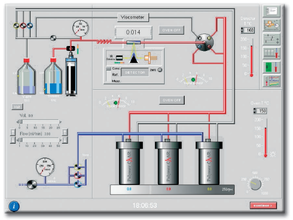
The Cryst-EX equipment is built in an Agilent GC oven where three stirred stainless steel vessels are installed. Samples are introduced manually in the crystallization vessels through a small cap before the automated analysis is initiated.

First the vessels selected are filled with preheated solvent (trichlorobenzene) through a syringe dispenser and the temperature goes up for sample dissolution according to a preselected method. At this point aliquots of each vessel solution are sampled with the dispenser and taken to the Infrared detector cell to calculate the initial concentration (full dissolution).

The oven goes now to the low temperature setpoint to precipitate the insoluble fraction. After a certain time aliquots of each vessel solution are sampled through an internal filter to collect, only, the soluble fraction which is analyzed for concentration.

The Cryst-EX performs now an automatic cleaning of the vessels to be ready for next analysis.

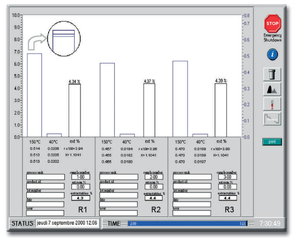
The whole process takes around 3 hours with no manpower intervention. The simplicity of the design with a dispenser, a single high temperature valve, a solid state IR detector and an electronic stirrer (no moving parts) makes the Cryst-Ex a very robust instrument for a QC Lab.



The concentration values at high and low temperature are shown, as obtained, in the computer screen and the ratio of the two concentrations multiplied by a calibration factor correspond to the equivalent Xylene Extractables. There is no need of accurately weighting the sample introduced as the ratio of the two concentration (full sample – soluble fraction) takes care of that.

The precision obtained with this method is very high as shown here with the results of a sample analyzed three times. A powder sample was analyzed 50 times in a QC industrial lab; The average Xylene extractables was 4.37% with a standard deviation of 0,08%.

In a QC laboratory a single vessel can be used to control the manufacturing process every 3 hours.



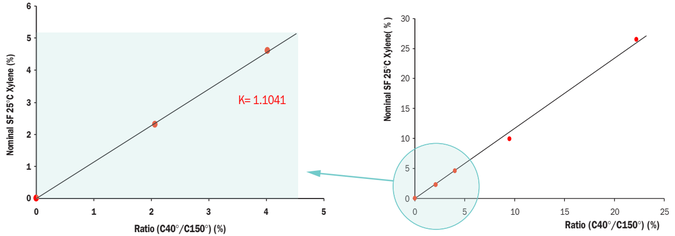
Xylene Extractables Calibration

The Cryst-EX was designed to be able to achieve fully equivalent data to the ISO 6427 method for Xylene extractables. In the development process it was decided, however, to experiment with a less flammable solvent (trichlorobenzene) provided that a calibration curve could be obtained, ideally linear, and covering a broad set of different PP copolymers as it is the actual case.

The use of trichlorobenzene, besides the safety aspects, would allow the concentration detection by Infrared which opens new possibilities as will be discussed later on for composition analysis. It was also decided to use a temperature of 40°C instead of the 25°C of the ISO method to avoid the need of subambient coolants in a QC laboratory.

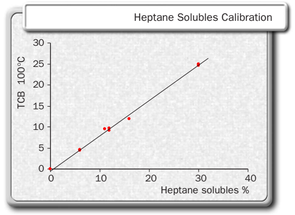
To calibrate the instrument a set of standards of well known Xylene %, at 25°C according to the ISO method is run on the Cryst-EX. A linear calibration as the one shown in the plot should be obtained covering a broad range of Xylene Extractables data for the type of PP copolymers of interest. The calibration coefficient (slope) is introduced in the software method to convert the TCB extractables at 40°C in Xylene extractables data at 25°C.

Cryst-ex



Heptane Solubles Calibration

The Heptane solubles according to the ISO method can be easily measured with the Cryst-EX apparatus by taking the same approach as with Xylene extractables. In this case the temperature to be used with TCB as solvent is 100°C and a good linear correlation is found between industrial samples analyzed by the ISO method and by Cryst-EX.

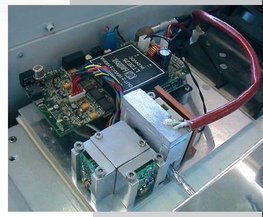


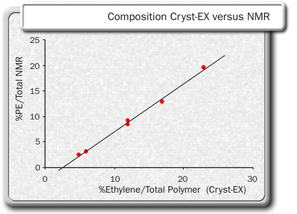
Incorporation of a Composition detector

The Cryst-EX instrument incorporates an OEM version of the new multiple wavelength infrared detector, IR4, and therefore it is capable of extending the standard 2 sensors up to 4 with all different wavelengths being measured simultaneously. An interesting application is to measure the CH3 and CH2 simultaneously in order to calculate the Ethylene percentage in a Polypropylene copolymer.

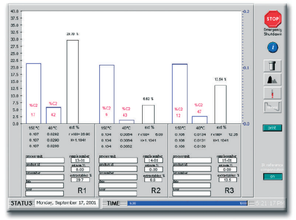
Most important is that the analytical process is not complicated at all, just new signals are obtained and processed.

The following table shows the analysis of a set of industrial samples analyzed with Cryst-EX and having the composition option. Besides the Xylene extractables data we obtain the Ethylene percentage in the original sample and in the Extractables fraction. The Ethylene % of the original samples as obtained by Cryst-EX correlates very well with NMR data on this set of samples as shown in attached plot.





	Original sample	Extractable fraction
Xylene Extract. (%)	Ethylene (%)	Ethylene (%)
25,4	23,2	52,9
17,8	12,2	38,9
8,6	5,1	26,4
26,6	16,6	41,9
5,9	5,8	42,8
12,1	12,1	46,5



The attached computer screen shows the extractables and composition data obtained in a set of three samples analyzed simultaneously.

Incorporation of Viscometer detector

The incorporation of a single capillary viscometer at the exit of the infrared detector in the way to waste allows, in a very simple maner and without complicating the analytical process, the measurement of the intrinsic viscosity (calculated as specific viscosity) of the original sample as well as the one of the extractable material.

In attached table three Polypropylene copolymers of different Melt Index are characterized by Cryst-EX with a single capillary viscometer. The intrinsic viscosities of the original material and the one of the Extractables fraction are shown.

As a summary Cryst-EX equipment can be extended with no significant cost with the composition and viscosity accessories. Quite a lot of information can be obtained automatically from a simple analysis, and most important the incorporation of these accessories does not reduce the robustness of the instrument.

	Sample A	Sample B	Sample C
MI	1,00	12,00	50,00
Extractables (%)	19,70	26,50	15,10
[h] Total sample	0,69	0,47	0,34
[h] Ext. Fraction	1,16	0,60	0,59