Determination of xylene-soluble fraction of polypropylene by Spin Track NMR Analyzer

GENERAL NOTES

Polypropylene (PP) is a synthetic polymer widely used as raw material in many industries. As for most of other polymeric substances, the reactivity of PP is in the direct dependence of accessibility of its free surfaces and volumes, i.e. it grows up with the increase of amorphous content. As established for decades, the chemical reaction and modification ability of PP is being attributed to estimation of the xylene-soluble fraction (XS).

Traditional chemical method of specified parameters determination (ASTM D5492-17 or ISO 16152) is based on solvent extraction. It requires dissolution of polymer in hazardous reagents at high temperature. This method is characterized by long analysis duration; results depend on personnel accuracy and experience. Moreover, a significant amount of operations is required for a single measurement.

The xylene-soluble fraction of polypropylene can also be estimated by Fourier transform infrared spectroscopy (FTIR). This technique reduces the measurement time, but it demands a thorough sample preparation. Noticeable, FTIR is mainly effective in studies of samples surface sometimes missing structural properties within bulk volume.

As an alternative to mentioned above methods, Time-Domain Nuclear Magnetic Resonance (TD-NMR) is becoming far more attractive. It provides quick measurements and acquires information out of the whole sample volume.

BASICS OF THE METHOD

Polypropylene consists of crystalline and amorphous phases. In the process of its dissolution in xylene, a partial transition of its amorphous phase into solution occurs. Accordingly, the proportion of amorphous phase in a sample of polypropylene correlates with the desired solubility of polymer in xylene.

The difference between crystalline and amorphous phases of polypropylene is observable in TD-NMR transverse relaxation decay, because they possess different relaxation times, so it's possible to derive the amorphous phase contribution to the signal. Hence this fact serves as the basis for an accurate and nondestructive technique, which is easily adaptable to on-line measurement and does not require any chemical reactions.

Measurements are based on exciting of sample nuclei by 90° radiofrequency pulse and subsequent detection of Free Induction Decay (FID) (fig.1). The signal from crystalline phase of polypropylene (A_{cr}) decays much faster than from amorphous part (A_{am}). The xylene-soluble fraction of polypropylene (XS) is proportional to the ratio $A_{am}/(A_{cr}+A_{am})$.

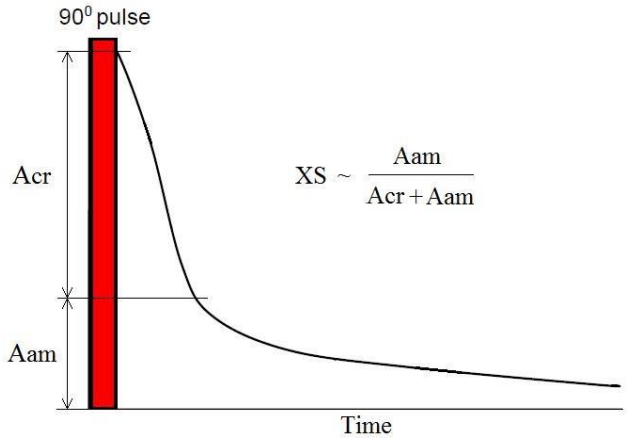

Fig.1. FID sequence

INSTRUMENTATION

NMR analyzer Spin Track (fig.2) from Resonance Systems is an ideal instrument for determination of Xylene-soluble fraction of polypropylene because of short probe ringing time, high acquisition rate and high signal to noise ratio which make measurements very reproducible and accurate.

Fig.2. Spin Track NMR Analyzer

Spin Track is built utilizing up-to-date electronics and sophisticated signal processing, it is very simple to use because all measurements are made automatically by pressing one button or inserting sample. **Spin Track** is able to be configured for remote control across via the Internet. The process can be organized as operator-free by using autosampler.

Application of the full "Xylene-soluble fraction of polypropylene" package allows producing exact and quick measurements with minimal costs.

The **XS Analyzer** package comprises:

- Spin Track NMR Analyzer with thermally stabilized magnetic system (induction 0.4 - 0.5 T with gap for 10 or 18 mm test tubes);
- PC with pre-installed Microsoft OS © Windows 7, 8, 10 or 11* and Relax 8 software;
- Thermostat "ST-80";
- Test tubes with outer diameter 10 or 18 mm**;
- Plastic caps for test tubes;
- Installation Manual;
- Method Sheet;
- Autosampler (optionally).

* Determined by the PC manufacturer
**Depends on the Spin Track configuration

CALIBRATION AND MEASUREMENT

The workflow consists of the following steps:

1. Filling sample tube;
2. Conditioning at 40°C for 20 minutes;
3. Inserting sample tube in a detector manually or by the autosampler;
4. Running a measurement which is taking few minutes;
5. All measurement results are recorded in a spreadsheet, saved and can be accessed both on a computer and on-line.

No weighing is required since the ratio of liquid protons to total protons is measured.

For calibration/validation stable polypropylene samples with the known xylene-soluble fraction can be used as well as the standards provided by Resonance Systems.

Calibration curve obtained by Spin Track Analyzer is shown below (fig.3).

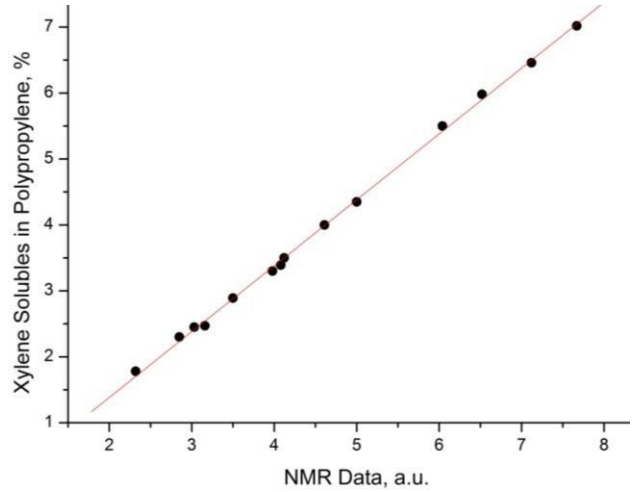

Fig.3. Typical calibration curve of Spin Track analyzer

FEATURES AND BENEFITS

- The Spin Track TD-NMR analyzer is noticeably affordable in the market of analytical instrumentation;
- Guarantee of reliable reproducibility by OEM;
- Requires no weighting of samples;
- Zero reagents costs and reduction in the number of operations performed by staff;
- Ability to determine the xylene-soluble fraction of polypropylene in the whole range of values;
- Process can be fully automated;
- 24/7 technical support.

CONTACTS

Please refer to additional information on the website of Resonance Systems **www.nmr-design.com**

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