

'Xylene solubles' (XS) is a historically established term denoting the percentage of soluble species in polypropylene homo- and co-polymers. This value is approximately proportional to the amorphous content of the materials. In practice this measure is widely used for product quality control and monitoring physical properties of the polymer during synthesis and processing.

Method

The xylene extraction method (ASTM D5492, technically equivalent to the standard ISO 16152) is commonly employed to determine the xylene solubles content by weight. However, this method requires dissolution of the products in a harmful solvent, use of high temperatures (135 and 150°C), large sample sizes (to guarantee reliable reproducibility), highly skilled analysts, and long measurement times.

The xylene solubles can also be measured using a Fourier transform infrared technique (FTIR). Although this method reduces significantly the analysis time, it requires delicate sample preparation and a highly skilled operator.

In contrast to the standard wet chemistry method and FTIR technique, low resolution Nuclear Magnetic Resonance (NMR) provides a fast, direct and user friendly method for determination of the xylene solubles content in polypropylene products that can be operated by a non NMR expert user. This technique is based on measurement of the NMR response obtained from amorphous part of the material, and quantification of the xylene solubles content by calibration.

Advantages of benchtop NMR

- NMR is very stable over the long-term, therefore requires little recalibration
- The sample measurement time is short (typically 20 seconds)
- Minimal sample preparation is required¹
- The NMR technique is non-destructive, so repeatability measurements can be made conveniently.

¹ Samples require preconditioning at 60°C for 20 minutes.



Calibration and Results

The calibration is generated using at least 6 samples of real products with predefined xylene solubles values spanning the range of concentrations of interest. Figure 1 shows a calibration for polypropylenes with xylene solubles content ranging from 0.9 to 4.9 % by weight (wt.-%). As seen in this figure, NMR gives an excellent linear correlation between the NMR response and the concentration of xylene solubles in the products.

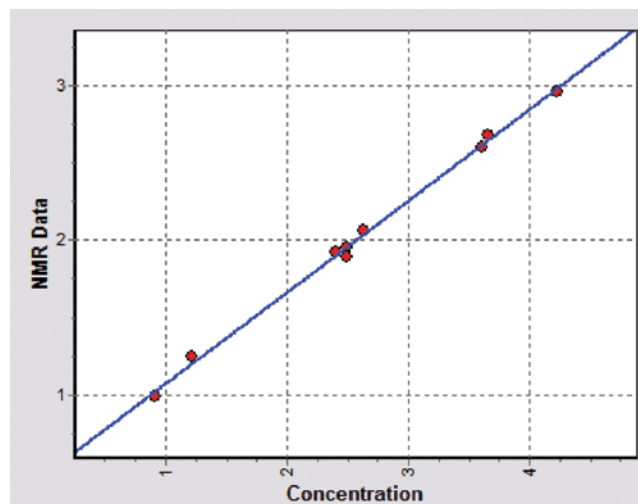


Figure 1: Calibration obtained for xylene solubles in polypropylenes; standard deviation of the linear fit is 0.06 wt.-%, correlation coefficient $R^2 = 1.00$. Measurements were made using Oxford Instruments MQC-23 benchtop NMR analyser fitted with an 18 mm diameter probe.

Table 1. Results of instrument repeatability test

Given XS Value, wt.-%	Results (wt.-%) of Repeat Measurements										Mean XS Value, wt.-%	Standard Deviation wt.-%
0.92	0.94	0.87	0.90	0.94	0.92	0.91	0.93	0.89	0.92	0.91	0.91	0.02

The instrument repeatability was tested by measuring one sample ten times. After every test measurement, the sample was transferred from the magnet bore back to the conditioning block for 20 min to be conditioned at 60°C and then measured again. Table 1 shows the repeatability test results.

Recommended Instrument Configuration

The **MQC-23** with 0.55 Tesla magnet fitted with an 18 mm diameter (8 ml) probe is ideal for this application. The 'Xylene Solubles in Polypropylene' package comprises:

- The **MQC-23** which can be controlled using its own built-in computer using Microsoft® Windows® or via a stand-alone PC
- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Xylene Solubles in Polypropylene' application
- Test / tuning sample
- Sample tubes with an outer diameter of 18 mm
- Polytetrafluoroethylene (PTFE) stoppers
- Stopper removal tool
- Installation Manual
- Method Sheet

In addition to this package the following equipment is required:

- Dry block heater capable of maintaining samples at 60°C
- Aluminium block with holes for 18 mm diameter tubes

The instrument offers multiple advantages over other instruments on the market:

- High signal sensitivity
- Small benchtop footprint
- Specific 'Xylene Solubles in Polypropylene' Applications Software
- Low maintenance
- Minimal sample preparation

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