

Summary

- Up to 250 times faster than wet chemistry methods
- No hazardous solvents required; no hazardous waste produced
- Easiest, most reliable technique available – suitable for unskilled personnel
- Reliable measurements down to low concentrations
- Ideal for high sample throughput

Application

In the production of artificial fibres such as polyamide and polyester, the fibres are sprayed with an oil-based coating to reduce static electricity and friction as well as enhance certain physical characteristics. This coating is variously known in different countries as spin finish, oil pick-up (OPU) and finish on yarn (FOY). Measurement of the applied spin finish using the **MQC** analyser is fast, simple and solvent free. Like its predecessor the MQA, the **MQC** supports a non-weighing method which allows even faster measurements. The fast, precise results obtainable with the **MQC** allow tighter control of the manufacturing process which translates, in real terms, to fewer out of specification products and lower production costs due to less finish material being used.

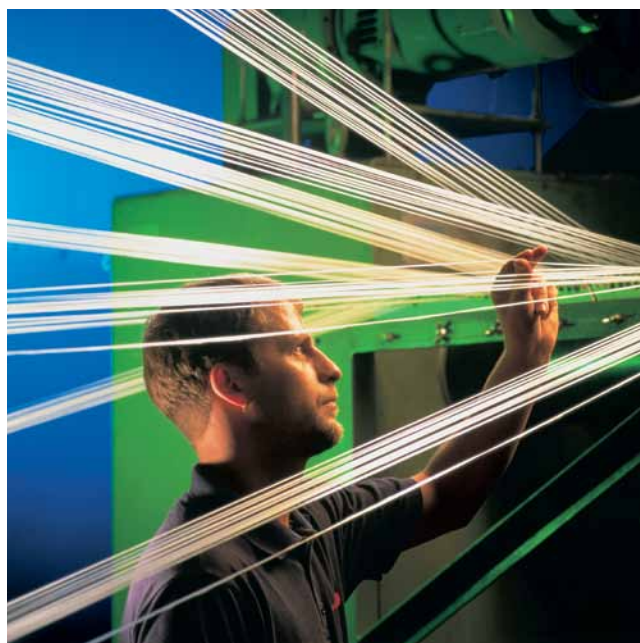
Advantages of NMR

The traditional method of testing is to dissolve the coating in an organic solvent and then determine the amount of dissolved oil in the solvent either gravimetrically (following distillation) or by use of infrared spectroscopy. All these methods are time consuming, use hazardous solvents and require skilled operators. Benchtop NMR provides an alternative method which is quick and easy to perform, simple to calibrate, and capable of determining finish levels below those accurately measurable by solvent extraction.



Method

Benchtop NMR detects the hydrogen signal of oil after the signal from the solid fibre has decayed. Dividing this signal by mass gives the hydrogen density of oil on the fibre which correlates with the spin finish content. For some samples it is possible to substitute mass with the total NMR signal from oil and fibre thus removing the necessity to weigh the sample. This is often called the ratio or non weighing method.



Weighing Method

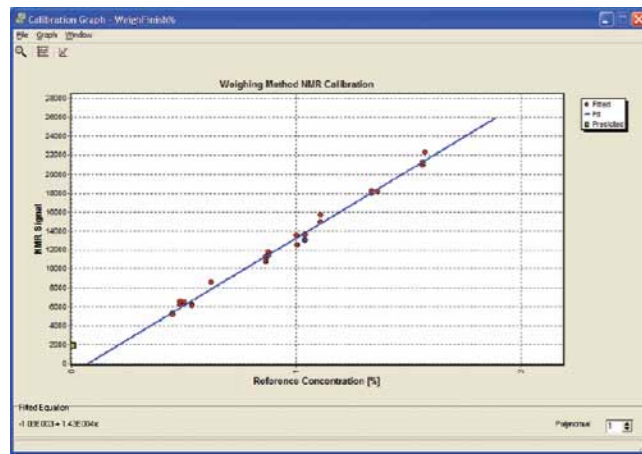


Figure 1: Calibration curve for the Weighing method produced by the MQC (SD = 0.0325, r² = 0.9962)

Calibration

Ultimately only two well known standards are required to calibrate the instrument. However, initially it is recommended that the instrument is calibrated by 3-6, preferably more, standards with known coating weights evenly spread over the range of interest. NMR is a comparative technique therefore its accuracy is only as good as that of the reference technique against which it is being compared.

Thereafter Artificial Setting Up Samples (SUSs) are available for restandardising calibrations to compensate for small drifts in the instrument, thus improving performance and ease of use. Given that SUSs are very stable, they can be used in the long term thus avoiding having to recalibrate an instrument on a regular basis using real samples.

Measurement

Samples are first weighed (weighing method only), pushed into a sample tube, then compressed to the optimum height using a PTFE stopper. After a suitable conditioning time, either at room temperature or 40°C, the sample is inserted into the instrument. Measurement time is approximately one minute per sample.

Weights can either be entered manually or transferred automatically from the electronic balance into the application software (weighing method at room temperature only).

| Sample Name | Given Spin Finish Content | Measured Finish Content | Difference |
|-------------|---------------------------|-------------------------|---------------|
| 1 | 1.335 | 1.330 | -0.006 |
| 1 | 1.335 | 1.344 | 0.009 |
| 1 | 0.877 | 0.869 | -0.008 |
| 1 | 0.877 | 0.897 | 0.020 |
| 1 | 0.486 | 0.506 | 0.020 |
| 1 | 0.486 | 0.531 | 0.045 |
| 2 | 1.565 | 1.536 | -0.029 |
| 2 | 1.565 | 1.559 | -0.006 |
| 2 | 0.537 | 0.509 | -0.028 |
| 2 | 0.537 | 0.500 | -0.037 |
| 2 | 1.039 | 0.984 | -0.055 |
| 2 | 1.039 | 1.026 | -0.013 |
| 3 | 0.450 | 0.440 | -0.010 |
| 3 | 0.450 | 0.433 | -0.017 |
| 3 | 0.864 | 0.828 | -0.036 |
| 3 | 0.864 | 0.862 | -0.002 |
| 3 | 1.110 | 1.170 | 0.060 |
| 3 | 1.110 | 1.117 | 0.007 |
| 4 | 0.623 | 0.674 | 0.051 |
| 4 | 1.005 | 0.952 | -0.053 |
| 4 | 1.364 | 1.347 | -0.017 |
| 5 | 1.575 | 1.637 | 0.062 |
| 5 | 0.503 | 0.528 | 0.025 |
| 5 | 1.004 | 1.020 | 0.016 |
| | | Average | 0.000% |
| | | Std. Dev. | 0.033% |

Table 1: Comparison of NMR vs. given spin finish contents using the weighing method on the MQC

Results

Figures 1 and 2 show the calibration graphs produced, and Tables 1 and 2 show the results from the same calibration samples measured, using weighing and non-weighing methods respectively.

Both graphs show good correlations between the reference values supplied and the NMR signal, r² = 0.996 and 0.995 respectively for the weighing and non-weighing methods. They also show that the spin finish can be measured accurately

Non Weighing Method

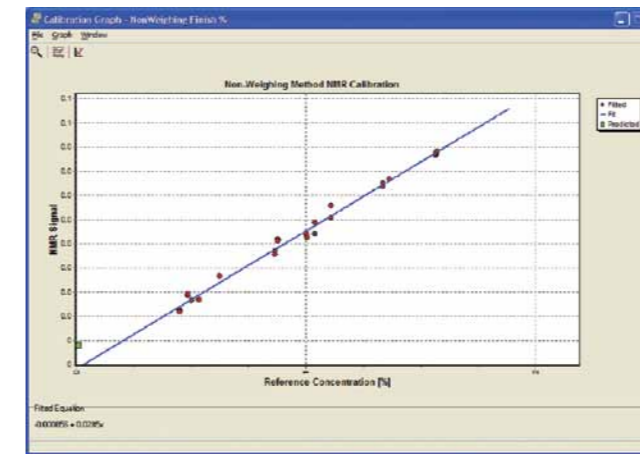


Figure 2: Calibration curve for the non-weighing method produced by the MQC (SD = 0.0375, r² = 0.9949)

by NMR with standard deviations of 0.033% and 0.038% between the given and measured values for the weighing and non-weighing methods respectively. Of the two methods, the weighing method yielded fractionally better correlation and accuracy than the non-weighing method; however, the non-weighing method has the advantage of reducing the amount of work required to carry out the analysis.

Conclusion

- NMR is very stable over the long term and rarely needs calibration adjustment. If required, this can be done simply using stable Setting Up Samples which recreates the original calibration carried out during installation
- NMR penetrates through the whole sample and is insensitive to air voids, which means it provides the most accurate measurement of the total amount of oil in a given volume of sample
- In general, NMR is insensitive to colour
- The NMR technique is non-destructive so the same sample may be measured several times before being analysed by other techniques
- Sample measurement time is rapid (typically 64 seconds)

| Sample Name | Given Spin Finish Content | Measured Finish Content | Difference |
|-------------|---------------------------|-------------------------|---------------|
| 1 | 1.335 | 1.326 | 0.009 |
| 1 | 1.335 | 1.347 | -0.012 |
| 1 | 0.877 | 0.940 | -0.063 |
| 1 | 0.877 | 0.928 | -0.051 |
| 1 | 0.486 | 0.529 | -0.042 |
| 1 | 0.486 | 0.540 | -0.054 |
| 2 | 1.565 | 1.548 | 0.017 |
| 2 | 1.565 | 1.559 | 0.006 |
| 2 | 0.537 | 0.497 | 0.040 |
| 2 | 0.537 | 0.499 | 0.038 |
| 2 | 1.039 | 0.979 | 0.060 |
| 2 | 1.039 | 1.063 | -0.024 |
| 3 | 0.450 | 0.424 | 0.026 |
| 3 | 0.450 | 0.411 | 0.039 |
| 3 | 0.864 | 0.828 | 0.036 |
| 3 | 0.864 | 0.853 | 0.011 |
| 3 | 1.110 | 1.185 | -0.075 |
| 3 | 1.110 | 1.095 | 0.015 |
| 4 | 0.623 | 0.670 | -0.047 |
| 4 | 1.005 | 0.957 | 0.048 |
| 4 | 1.364 | 1.373 | -0.009 |
| 5 | 1.575 | 1.575 | 0.000 |
| 5 | 0.503 | 0.492 | 0.011 |
| 5 | 1.004 | 0.980 | 0.024 |
| | | Average | 0.000% |
| | | Std. Dev. | 0.038% |

Table 2: Comparison of NMR vs given spin finish content using the non-weighing method on the MQC



Oxford Instruments Ready-to-Run Application Package

For accurate determination of low spin finish levels, the **MQC-23** with a 0.55 Tesla (23 MHz) magnet, fitted with an 18 mm diameter (8 ml sample) probe is ideal. The Spin Finish on Fibre package comprises:

- **MQC-23** which can be controlled using its own built-in computer operating Microsoft Windows or via a stand-alone PC
- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Spin Finish' application
- Test/tuning sample
- 18 mm glass tubes

- PTFE stoppers (to seal the tubes)
- Stopper insertion/removal rod
- Installation manual
- Method sheet

In addition you may require:

- A dry block heater and aluminium block with holes for sample conditioning at 40°C
- A precision balance (weighing method only)
- A set of four Spin Finish Setting-Up-Standards (SUSs)



Oxford Instruments Magnetic Resonance

For more information please email:
magres@oxinst.com

UK

Tubney Woods, Abingdon
Oxon, OX13 5QX, UK
Tel.: +44 (0) 1865 393 200
Fax: +44 (0) 1865 393 333

China

Room 1/E, Building 1
Xiangzhang Garden
No. 248 Donglan Road
Shanghai 201102, China
Tel: +86 21 6073 2925
Fax: +86 21 6360 8535

USA

300 Baker Avenue, Suite 150
Concord, Mass 01742, USA
Tel: +1 978 369 9933
Fax: +1 978 369 8287

visit www.oxford-instruments.com for more information

www.oxford-instruments.com

This publication is the copyright of Oxford Instruments and provides outline information only which (unless agreed by the company in writing) may not be used, applied or reproduced for any purpose or form part of any order or contract or be regarded as a representation relating to the products or services concerned. Oxford Instruments' policy is one of continued improvement. The company reserves the right to alter, without notice, the specification, design or conditions of supply of any product or service. Oxford Instruments acknowledges all trademarks and registrations. Microsoft and Windows are registered trademarks of the Microsoft Corporation in the United States and other countries.
Ref. SF-08-11



The Business of Science®

