

# Measurement of Spin Finish on Artificial Fibre using the Weighing and Non-Weighing Methods

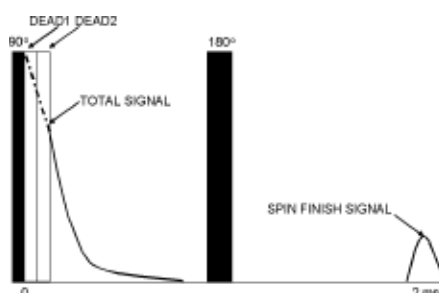
In the production of artificial fibres such as polyamide and polyester, the fibres are sprayed with an oil-based coating to reduce static electricity and friction as well as enhance certain physical characteristics. This coating is variously known in different countries as spin finish, oil pick-up (OPU) 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.

## Method

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. Some variations also require the use of mercury-containing catalysts. Low resolution pulsed 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.

In general, the NMR method consists of correlating the spin finish against the HAHN-echo amplitude (at 2 ms echo time) divided by the sample mass. For some samples it is possible to substitute the Total Signal, obtained from the Free Induction Decay (FID), for the mass and correlating this value with the spin finish content

thus removing the necessity to weigh the sample. This is often called the ratio or non-weighing method.

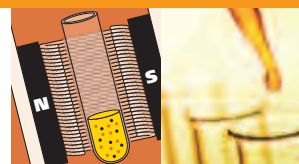


The high speed digital sampling and wide dynamic range of the MQC mean that the MQC can achieve excellent results from the non-weighing method as shown by the results below.

## Calibration and Results

It is recommended that the instrument is calibrated by measuring a set of at least 6, preferably 12, standards with known coating weights evenly spread over the range of interest.

In the standard (weighing) method, samples are first weighed, 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 be entered manually or transferred automatically from electronic balance into the application software (weighing method only).



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# Weighing Method

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,  $r^2 = 0.996$  and  $0.995$  respectively for the weighing and non-weighing methods. They also show that the spin finish can be measured accurately 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.

Artificial Setting Up Samples (SUS's) are available for restandardising calibrations to compensate for small drifts in the instrument, thus improving performance and ease of use. Given that SUS's 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.

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
<b>Average</b>			<b>0.000%</b>
<b>Std. Dev.</b>			<b>0.033%</b>

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

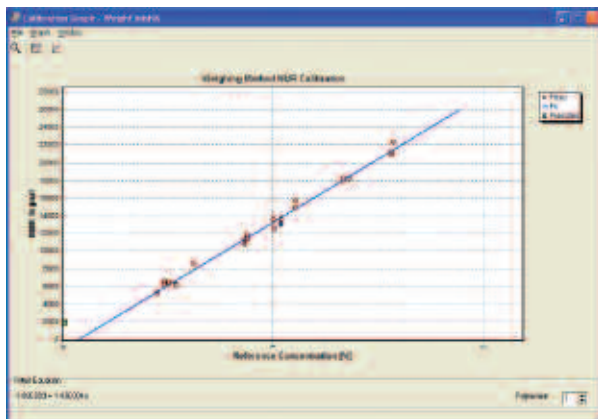


Figure 1. Calibration curve for the Weighing method produced by the MQC. (SD = 0.0325,  $r^2 = 0.9962$ )

# Non Weighing Method

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
		<b>Average</b>	<b>0.000%</b>
		<b>Std. Dev.</b>	<b>0.038%</b>

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

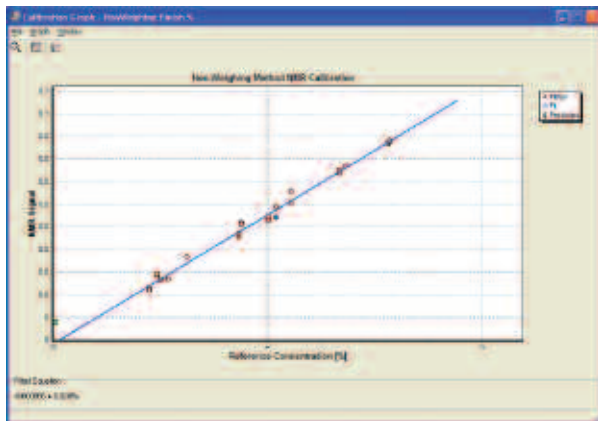


Figure 2. Calibration curve for the non-weighing method produced by the MQC (SD = 0.0375,  $r^2 = 0.9949$ )

## Recommended Instrument

For accurate determination of low spin finish levels, the MQC-23 fitted with an 18 mm diameter (8 ml sample) probe is a suitable instrument for this application. The Spin Finish package comprises:

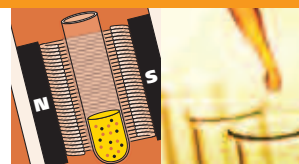
- **MQC-23** with a computer incorporated, operating the latest version of Microsoft Windows (no separate PC is required)
- **MultiQuant** software including **RI Calibration, RI Analysis**, and the EasyCal 'Spin Finish' application.
- 18 mm glass tubes.
- PTFE stoppers (to seal the tubes).
- Stopper insertion/removal rod.
- Installation manual.
- Method sheet.

In addition to this package you may also wish to purchase:

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

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

- High signal sensitivity
- Small benchtop footprint
- Low maintenance
- The sample tubes are recyclable, so there are no consumables
- Minimal sample preparation



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