

Measurement of Spin Finish on Artificial Fibre



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 other physical characteristics. This coating is known in different countries as spin finish, oil pick-up (OPU) and finish on yarn (FOY). Measurement of the applied spin finish by low resolution Nuclear Magnetic Resonance (NMR) is fast, simple and solvent free and allows tighter control of the manufacturing process. This 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 infra-red 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 is capable of determining finish levels below those accurately measurable by solvent extraction. For instance, the measurement of mineral oil on staple polyester is particularly difficult because of the low levels of finish and therefore is an ideal application for NMR.

Calibration and Results

After setting the relevant method parameters, a calibration must be generated before unknown samples can be measured. This is done by measuring a set of standards of known coating weight using the **EasyCal** calibration software.

Samples are weighed, and pushed into a sample tube, then inserted into the instrument. Measurement time is approximately one minute per sample. Weights can be entered manually or automatically from an electronic balance, into the application software.

It is recommended that at least six, preferably 12, calibration standards should be used, with coating weights evenly spread over the range of interest. When all the standards have been measured, the calibration can be edited on-screen using the **RI Calibration** software. Calibrations can be stored on disk and recalled later for editing or to add extra measurement points. If reference values are not known at the time of measurement, they can be added later if the file has been saved to disk.



Sample set 1

Sample	Ref.	NMR 1	NMR 2
1	0.06	0.06	0.06
2	0.13	0.13	0.14
3	0.22	0.23	0.22
4	0.31	0.30	0.30
5	0.39	0.38	0.38
6	0.48	0.47	0.47
7	0.58	0.57	0.57
8	0.67	0.67	0.67
9	0.75	0.76	0.76

Sample set 2

Sample	Ref.	NMR 1	NMR 2
1	0.74	0.73	0.73
2	0.57	0.58	0.58
3	0.49	0.49	0.49
4	0.45	0.46	0.46
5	0.41	0.40	0.40
6	0.38	0.36	0.36
7	0.33	0.35	0.35

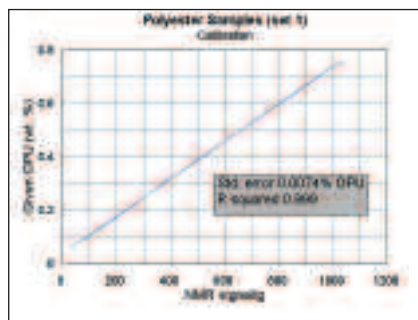
Comparison of NMR versus Extraction/Infra-Red

Extraction/Infra-Red	NMR	Difference
0.74	0.72	0.02
0.57	0.60	-0.03
0.53	0.53	0.00
0.49	0.49	0.00
0.45	0.47	-0.02
0.41	0.41	0.00
0.36	0.35	0.01
0.33	0.33	0.00
0.35	0.36	-0.01

Results are shown here from four separate sets of samples – two polyester, one polyamide and one nylon-based fibre.

Sample set 1 (polyester)

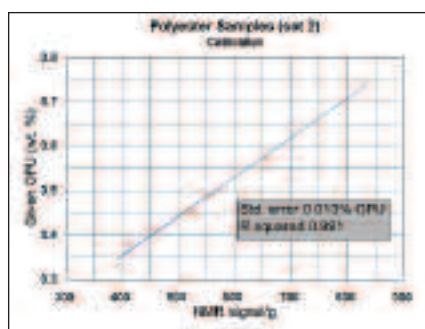
A set of nine calibration standards between 0.06% and 0.75% finish (water/oil emulsion) were measured twice. The two sets of results are shown opposite.



Sample set 2 (polyester)

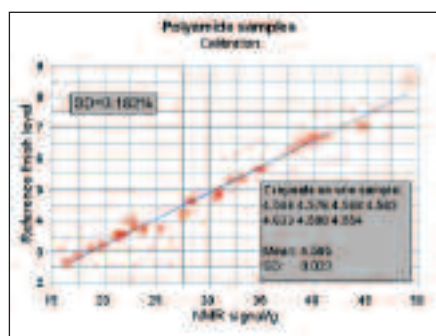
A set of 7 calibration standards between 0.33% and 0.74% finish (water/oil emulsion) were measured twice, with the results shown opposite.

A further nine samples were measured using the calibration produced using sample set 2 and compared against values determined by solvent extraction followed by infra-red assessment of oil in the solvent. The results are shown opposite.



Sample set 3 (polyamide)

The polyamide set comprised 31 calibration standards of which six were a very poor fit to the calibration line and were deleted. The finished calibration was as shown below. One sample was then measured seven times in succession to determine the repeatability which was shown to be 0.023.



Sample set 4

Sample	Ref.	NMR 1	NMR 2	Difference
1	0.87	0.89	0.88	0.00
2	1.06	1.04	1.04	-0.01
3	1.20	1.16	1.15	0.00
4	1.21	1.24	1.25	-0.01
5	1.35	1.39	1.39	0.00
6	1.46	1.37	1.38	-0.02
7	0.72	0.79	0.79	-0.01
8	1.20	1.18	1.19	-0.01
9	0.96	1.07	1.08	-0.01
10	1.34	1.31	1.30	0.00
11	1.52	1.46	1.46	0.00
12	1.04	0.96	0.97	-0.01
13	0.85	0.81	0.82	-0.01
14	1.55	1.63	1.64	-0.01

Sample set 4 (nylon)

This set comprised of 14 calibration standards between 0.72% and 1.55% finish (water/oil emulsion). There was some doubt about the accuracy of the reference values, so the NMR repeatability was the main interest.

The instrument repeatability was obtained using one polyester sample measured 10 times, removing and re-inserting the sample tube before each measurement. Instrument repeatability was shown to be 0.001 (at the 0.375% level).

The full set of 14 samples were run twice, the results of which are shown at the top of the next column.

Recommended Instrument

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

- **MQC-23** with a built-in computer 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
- A set of three Spin Finish Setting-Up-Standards (SUSs)
- 18 mm glass tubes
- PTFE stopper (to seal the tubes)
- Stopper insertion/removal rod
- Installation manual
- Method sheet

In addition to this package you will also require:

- A dry heater and aluminium block with holes for sample conditioning at 40°C
- A precision balance

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

- High signal sensitivity
- Small benchtop footprint
- Low maintenance
- The sample tubes are recyclable, lowering consumable costs
- Minimal sample preparation

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