# Measurement of fluorinated coatings on fibres and textiles

Application Note 27


**Summary**

- Fast, accurate and repeatable
- No sample preparation
- Simple linear calibration
- Easiest, most reliable technique available; suitable for non-technical personnel


## Application

Fluorinated coatings are often applied to fibres and textiles to modify their properties. For example, fluorocarbon-based finishes are often applied to carpet fibres to provide oil or water repellent properties. Furthermore, Teflon is applied to textiles to provide easy cleaning, water resistance and fire-retardant properties. Therefore, there is a requirement to measure the amount of fluorinated coating to optimise the process and, in turn, the properties of the product.

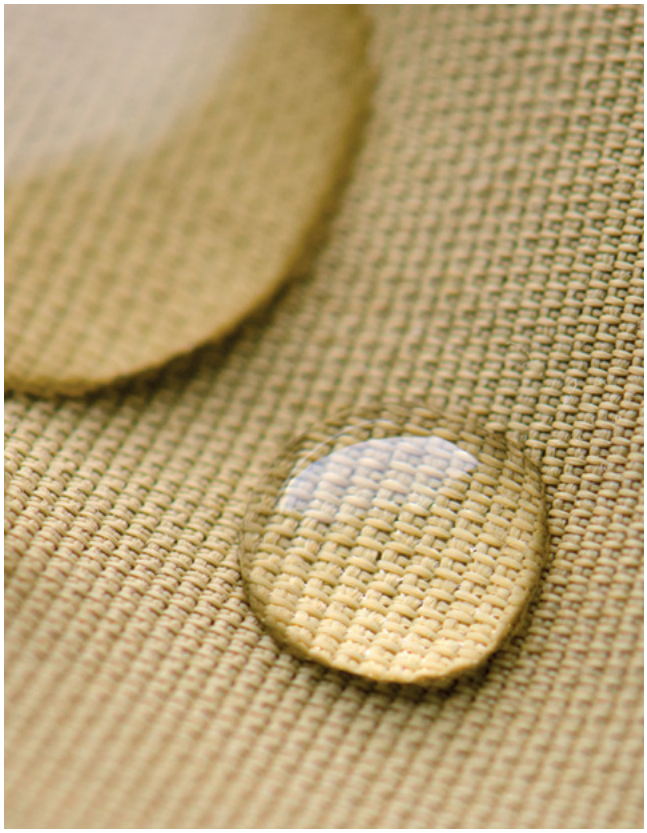
## Advantages of NMR

Conventional wet chemical methods used to determine fluorine content include ion selective electrode and ion chromatography. Both methods are time-consuming, require skilled chemists and involve the use of potentially hazardous chemicals which require disposal, all of which contribute to the cost of the analysis.

The **MQC+** time domain NMR analyser offers a rapid, easy and non-destructive method for measurement of fluorine on textiles which may be used for routine analysis in a production environment without any requirement for additional chemicals and specialist operator training. Using a sample holder tuned to the hydrogen frequency, it is also possible to measure finish removal prior to the addition of another coating.

## Method

The analytical technique is based on direct measurement of the Nuclear Magnetic Resonance (NMR) signal of fluorine-19 which has 100% natural abundance. The acquired NMR signal is normalised by the sample mass and then the fluorine content (weight-%) is calculated using an appropriate calibration curve.



## Calibration

It is possible to calibrate the **MQC+** using only two samples if the reference values are known to be accurate. However, it is recommended that initially the instrument is calibrated using 3-6, preferably more, standards with known fluorine contents, 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.

Figures 1 shows a calibration for fluorine content from a Teflon coating.

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## Measurements

A tared sample tube is filled to a given height with the fibre, or in this instance, textile sample, then weighed prior to NMR analysis. The samples are then conditioned at 40°C. Measurement time is 5 minutes per sample.

## Results

Table 1 shows that the NMR measurement is repeatable.

## Conclusions

- NMR is very stable over the long term and rarely needs calibration adjustment.
- Minimal sample preparation is required. The sample is measured "as is" without alteration.
- Measurement precision is good compared to wet chemical methods.
- Results can be obtained in minutes.
- The NMR technique is non-destructive so the same sample may be measured several times before being analysed by other techniques.
- With a different sample holder for measuring hydrogen, the NMR instrument may also be used for the measurement of other liquid coatings as well as finish removal.

***Figure 1.*** *NMR calibration for fluorine content. Correlation Coefficient 1.00; Standard Deviation 0.015%*

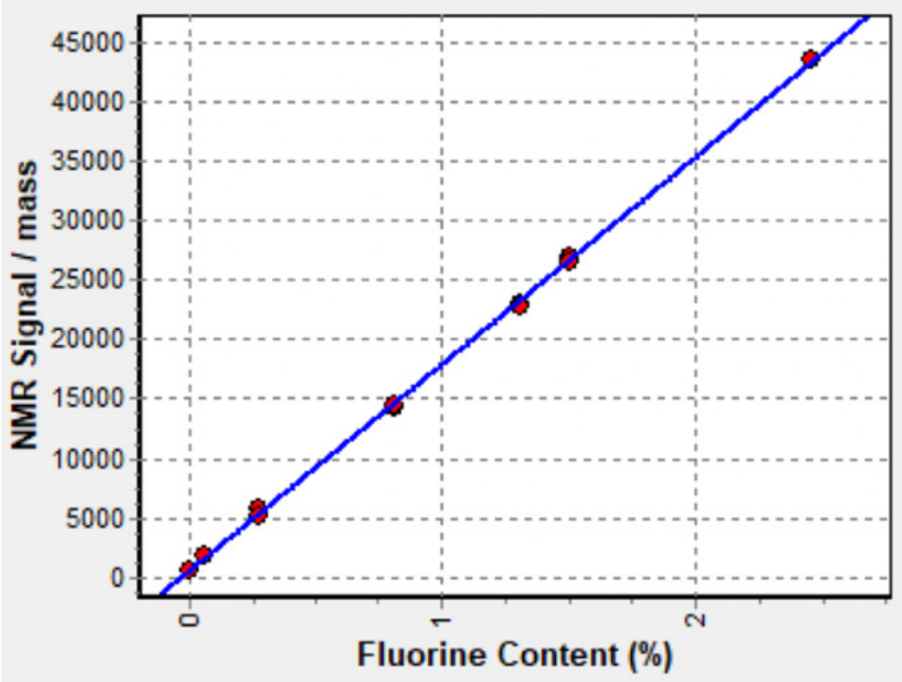


**Table 1: Results of measurement repeatability test**

| Repeat NMR measurements F-Content (Weight-%) | | | | | | | Mean F-Content (Weight-%) | Standard Deviation (Weight-%) |
|---|---|---|---|---|---|---|---|---|
| 0.69 | 0.68 | 0.70 | 0.73 | 0.68 | 0.69 | 0.72 | 0.70 | 0.02 |

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