Measurement of Limestone Filler in Asphalt

Summary

- Fast, accurate and repeatable
- Simple linear calibration
- Little sample preparation
- Easiest, most reliable technique available; suitable for unskilled operators – simple, intuitive visual software
- Non-destructive technique

Application

Asphalt shingles are one of the most prevalent roof coverings used in North America. They are a composite product, with a base layer of paper or fibreglass, which is impregnated or coated with an asphalt layer. This asphalt layer is itself a composite of asphalt (bitumen in British English) and finely divided mineral (often limestone) fillers. The performance and longevity of asphalt shingles is critically dependent on an accurate and consistent formulation of the asphalt layer.

Advantages of NMR

Several methods are available to determine the filler content of asphalt. Perhaps the most commonly used is the loss-on-ignition method which involves measuring the weight loss of a bituminous mixture during combustion in a furnace. This test can take 40-60 minutes for 'large' samples, can be hazardous and costly, because of the temperatures used, and produces hazardous fumes that must be directed out of the facility.

By contrast, NMR requires little sample preparation and the measurement is fast, repeatable and accurate. This technique is a turn key solution and does not require special operator training.

Method

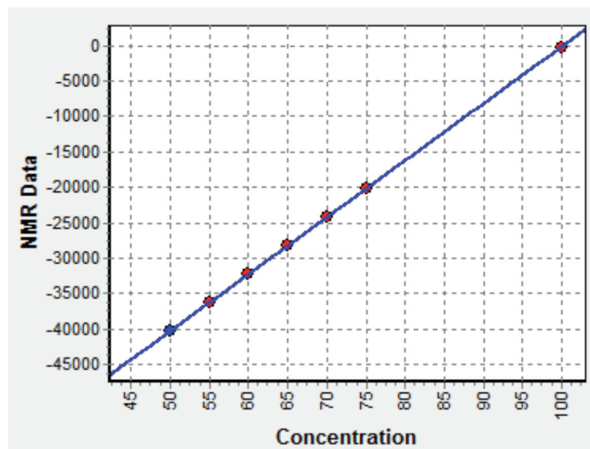
Benchtop NMR indirectly measures the filler content in asphalt by measuring the NMR signal (per unit mass) from the asphalt and subtracting the asphalt content from the total mass, to give the filler content. (%Filler = 100% - %Asphalt)

Calibration

The instrument is calibrated using a single sample of the asphalt thus can be easily recalibrated if the raw material regularly changes. The notional mass of the calibration sample is varied to give a range of filler contents and pure filler itself can be used as a 0% asphalt calibration point.

Measurement

A sample vial is tared then filled to a given height with the sample. Each sample is then weighed before placing in a temperature conditioning block for 20 minutes. The conditioning temperature is usually 40°C, the same as the magnet, for optimum precision.



Filler in Asphalt calibration graph

Results

Table 1 shows the excellent reproducibility obtained for artificially-created (50 % filled) and real (originally unknown filler content) sub-samples.

Sample	%Filler				
	Sub-sample 1	Sub-sample 2	Sub-sample 3	Average	Std Dev
50% filled	49.94	49.98	50.19	50.04	0.13
Real (Unknown)	65.27	65.08	65.46	65.27	0.19

Table 1. *Measurement reproducibility of different unfilled and filled coating samples*

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Table 2 shows the excellent repeatability obtained from multiple measurements of the filled sample. This reproducibility test on the same sample is only possible because NMR is a non-destructive technique, unlike the loss-on-ignition method, in which the test destroys the sample.

Repeat	% Filler
1	65.25
2	65.24
3	65.26
4	65.30
5	65.36
6	65.35
7	65.26
8	65.32
9	65.33
10	65.35
Mean	65.30
SD	0.05

Table 2. *Measurement repeatability on the same filled coating sample*

Conclusions

A primary calibration can be produced using a single sample of asphalt.

- NMR is very stable over the long term and rarely needs calibration adjustment
- Because NMR penetrates through the whole sample and is insensitive to air voids, it provides the most accurate measurement of filler in a given volume of sample
- The measurement precision is typically better than 0.1% filler
- Sample measurement time is rapid (32 seconds)*

* For optimal precision samples should be conditioned at 40°C for 20 minutes in a dry block heater prior to analysis.

Oxford Instruments Ready-to-run Application Package

The **MQC**23 with a 0.55 Tesla (23 MHz) magnet, fitted with a 26 mm diameter (10 ml sample) probe is ideal for this application. The **MQC**23 uniquely has the largest gap between magnet poles for a 0.5 (actually 0.55) Tesla magnet. This results in the desirable combination of largest sample size with the highest sensitivity. The Filler in Asphalt 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** 'Filler in Asphalt' application
- Test/tuning sample
- 23 mm glass vials
- PTFE sample vial holders
- 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

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