

Determination of Oil in Wax



Although wax was originally treated as a waste product by the oil refining industry, it is now widely used in manufacturing because of its specific mechanical and physical properties (flexibility, friction index, hardness coefficient, melting point etc.). The oil refining industry has responded by producing different types of wax with special characteristics (petrolatum, slack, crude paraffin, recrystallised paraffin, blend etc.). As oil content is one significant parameter in the composition of wax that affects its physical properties, it needs to be measured routinely.

Method

The official oil in wax method (American Society for Testing and Materials (ASTM) D721-06 IP158/69 (01)) involves dissolving the oil in methyl ethyl ketone, then precipitating the wax at low temperature (-35°C). The technique is slow (2 hours), requires technical skills and the use of hazardous solvents. In comparison, bench-top Nuclear Magnetic Resonance (NMR) offers rapid, user-friendly, safe and reliable analysis in response to the increasing demand for Quality Control (QC) in routine operation. The Oil in Wax NMR method is a ratio method so the sample does not even need to be weighed.

In general, the Oil in Wax NMR method can exploit two different hardware configurations to optimise for two ranges of oil content dependent on the sample and /or user requirements:

- 2% and above: rapid measurement using a standard **MQC** with 10mm diameter samples and sample pre-conditioning at low temperature (usually -5°C).
- 0.1% to 2% oil: low detection limit measurement using an **MQC** with 10mm diameter samples and equipped with a low-maintenance, fluid-cooled temperature-regulated probe (usually -5°C or less).

Although most types of wax should fall into one of these categories, our Applications Scientists can evaluate your wax products as part of our development policy. Experience shows that different types of wax may have different physical, and consequently NMR properties which may need to be optimised separately.

For example, in one set of wax samples, a calibration was obtained for the majority (Figure 1) using the reference values determined by the ASTM method. However a number of samples lay off this curve and were only otherwise distinguishable by their high melting point.

The accuracy of the samples with oil content < 2% could be further enhanced by generating a separate calibration using only those samples.

The **MultiQuant** software is versatile such that the calibrations can be optimised after the reference samples have been measured and they do not have to be measured again to produce different calibrations. However, it is recommended that the instrument is recalibrated regularly especially if new wax products need to be measured.

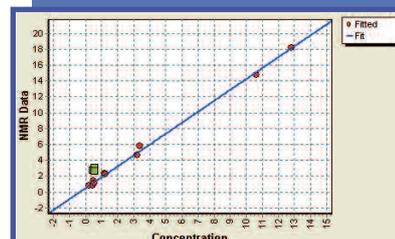


Figure 1. NMR signal ratio at -5°C versus oil content determined by the ASTM D721-06 method (SD 0.22, R 1.00). Analysis time 64 seconds. Three outliers labelled as green squares were removed for this calibration.

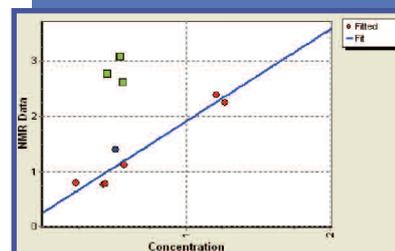


Figure 2. NMR signal ratio at -5°C versus oil content determined by the ASTM D721-06 method (SD 0.11, R 0.96). Analysis time 64 seconds. Same as Figure 1 but calibration generated from samples with oil contents <2%. Three outliers labelled as green squares were removed for this calibration.





NMR

Calibration and Results

The NMR technique requires calibration of the signal against the oil content (%) obtained by an appropriate reference method, e.g. solvent extraction technique (ASTM D721).

Calibration simply requires a minimum of six samples that span the concentration range of interest. Figure 3 shows the linearity and the accuracy of the NMR calibration curve for petrolatum in the oil range 10-40% as determined by the official ASTM method. Figure 4 demonstrates the precision and linearity of the calibration curve for hard wax at low detection limits, showing that measurements can be achieved below 1% oil (low detection limit method).

Figure 3. NMR signal ratio versus oil content in petrolatum. Analysis time ~ 20s. Mid-range measurement precision (95% confidence) ~ 0.1%. Calibration error ~ 0.1%.

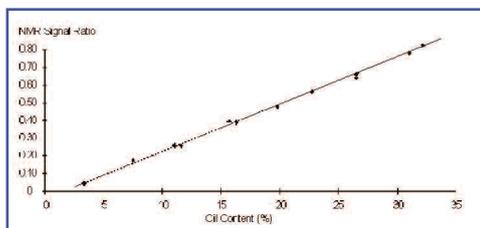
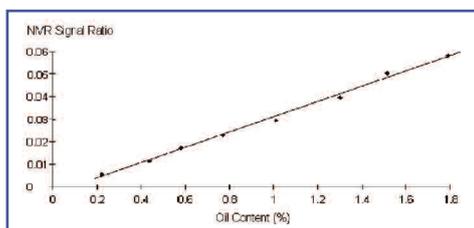


Figure 4. NMR signal ratio versus oil content in hard wax (low detection limit method only). Analysis time ~ 2 min. Mid-range measurement precision (95% confidence) ~ 0.05%. Calibration error ~ 0.07%.



Recommended Instrument

The **MQC-23** with a 0.55 Tesla (23 MHz) magnet is a suitable instrument for this application. There are two method configurations available:

For oil contents 2% and above:

- A standard 10 mm diameter (2 ml) hydrogen probe
- The method also requires a refrigerated water bath and aluminium block with holes for sample conditioning at low temperature (usually -5°C)

For oil contents 0.1% to 2%:

- A low maintenance 10 mm diameter (2 ml), fluid-cooled, temperature-regulated hydrogen probe
- The method also requires a refrigerated water bath with external circulating pump (for sample conditioning and control of the variable temperature probe) and an aluminium block with holes
- **Note:** a supply of dry air is also required for a temperature-regulated probe operating below ambient temperature to prevent condensation which will cause errors in the results

The Oil in Wax package also comprises:

- 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 'Oil in Wax'** application

- 10 mm diameter glass tubes
- Installation manual

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

- High signal sensitivity
- Low maintenance
- Recyclable sample tubes, lowering consumable costs
- Minimal sample preparation

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