Determination of Oil in Wax

Application Note 15

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)

** The Institute of Petroleum (IP) is now known as the Energy Institute.*

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 calibrated separately.

For example, Figure 1 shows the results for a set of waxes which were separated into three different calibrations dependent on the oil content and type of the wax. The MultiQuant software is versatile such that calibrations can be optimised after the reference samples have been measured and do not have to be measured again to produce different calibrations. It is good practice to recalibrate the instrument regularly, especially if new wax products need to be measured

Advantages of benchtop NMR

- NMR is a very stable technique over the long-term and therefore requires little re-calibration
- Minimal sample preparation is required
- No solvent extraction is required
- The NMR technique is non-destructive, so repeatability measurements can be made conveniently
- Sample measurement time is short (typically 1-2 minutes)

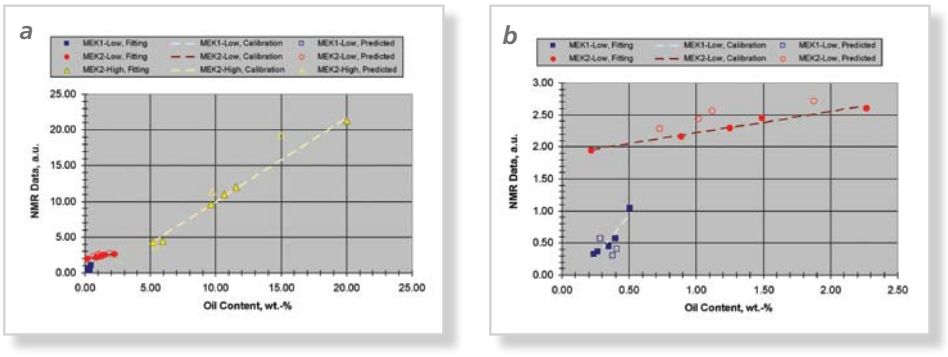

Figure 1: *Graphs showing calibration data for (a) all, and (b) low oil content, wax samples. Solid symbols represent data points accounted in calibrations and used for fitting; open symbols correspond to data points excluded from fitting to improve standard deviations and correlation coefficients of the calibration procedure; dashed lines are the calibration curves.*

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Figure 2: *NMR signal ratio versus oil content in petrolatum. Analysis time ~ 20s. Mid-range measurement precision (95% confidence) ~ 0.1%. Calibration error ~ 0.1%.*

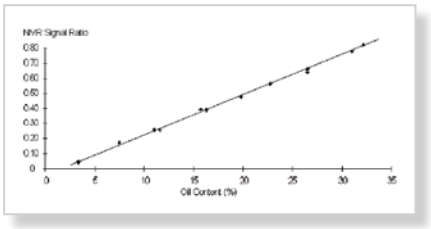
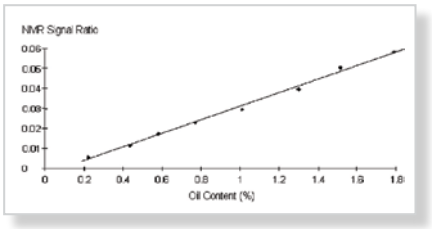


Figure 3: *NMR signal ratio versus oil content in hard wax (low detection limit method only). Analysis time ~ 2 min. Mid-range measurement precision (95% confidence) ~ 0.05%. Calibration error ~ 0.07%.*



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 (e.g. ASTM D721-06).

Ideally each calibration requires a minimum of six samples that span the concentration range of interest. Figure 2 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 3 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).

Recommended Instrument Configuration

The **MQC**-23 (0.55 Tesla) which can be controlled using its own Microsoft® Windows® or via a stand-alone PC and for oil contents 2% and above:

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

or for oil contents 0.1% to 2%:

- A low maintenance 10 mm diameter (2 ml), fluid-cooled, temperature-regulated hydrogen probe
- This 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:

- **MultiQuant** software including **RI Calibration, RI Analysis**, and the **EasyCal** 'Oil in Wax' application
- 10 mm diameter glass tubes
- Installation manual
- Method sheet

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

- High signal sensitivity
- Small bench-top footprint
- "Specific Oil in Wax" applications software
- Minimal sample preparation
- Low maintenance

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