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) D721-17, 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 uses the ratio of two signals so the sample does not even need to be weighed.

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)

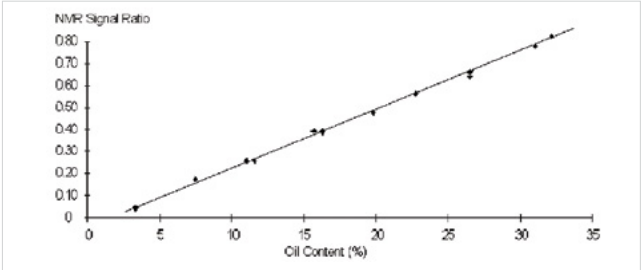

Figure 1: *NMR signal ratio versus oil content in petrolatum. Analysis time ~ 20s. Mid-range measurement precision (95% confidence) ~ 0.1%. Calibration error ~ 0.1%.*

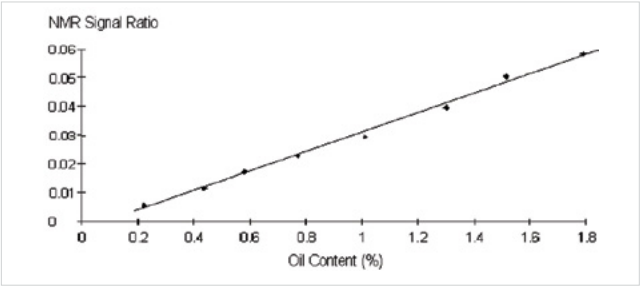

Figure 2: *NMR signal ratio versus oil content in hard wax (low detection limit method only). Analysis time ~ 2 min. Mid-range measurement precision (95% confidence) ~ 0.05%. Calibration error ~ 0.07%.*

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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 D721-17).

Ideally each calibration requires a minimum of six samples that span the concentration range of interest. Figure 1 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 2 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.

Recommended Instrument Configuration

The **MQC+** (0.55 Tesla) with a built-in computer operating Microsoft® Windows®10. This package also comprises:

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

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.

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

In addition, we can also provide:

- A refrigerated water bath with external circulating pump (for sample conditioning and control of the variable temperature probe)
- An aluminium block for 10mm tubes which fits in the above bath.

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

visit **www.oxford-instruments.com/mqc** for more information or email: **magres@oxinst.com**

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