Determination of grease content for water quality monitoring using the MQC23

Application Note 25

Summary

- Fast, accurate and repeatable analysis of low levels of grease content on a single filter sheet
- Minimal sample preparation
- Simple linear calibration
- Non-destructive technique, allowing solvent extraction verification after NMR analysis
- Easiest, most reliable technique available; suitable for non-specialised personnel
- Ideal for high sample throughput

Application

The measurement of grease content in waste water is a crucial parameter in water quality control and environmental monitoring performed by water suppliers' laboratories and environmental authorities. The analytical technique is targeted at fast, reliable and selective determination of grease content (mg) on dried filters following waste water filtration.

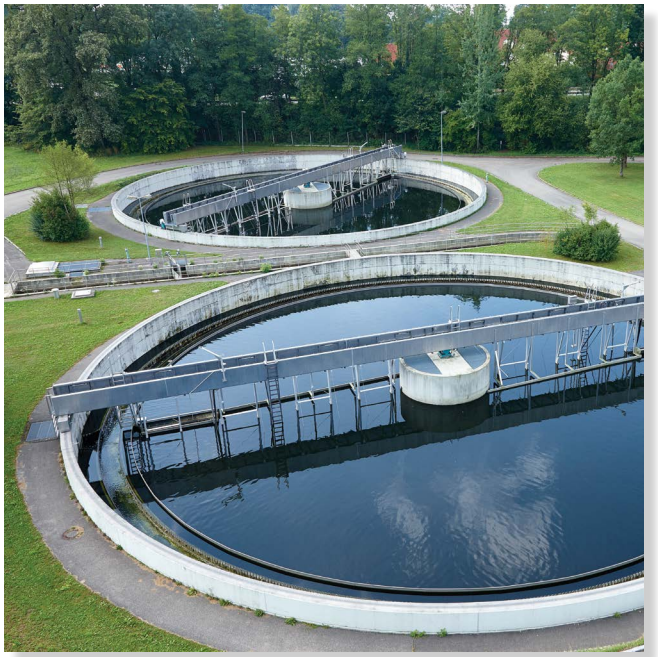
Advantages of NMR

Solvent extraction techniques are commonly used for determination of grease content on filters. However, these methods can be time consuming, require skilled operators and the use of hazardous solvents.

In contrast:

- NMR does not require solvents which are hazardous to use and in its disposal
- NMR is a bulk measurement technique and selectively measures all the oil and fat in the sample regardless of the distribution of the grease on the filter
- A linear calibration is readily generated using a few gravimetrically prepared samples
- NMR enables rapid measurement of different compositions of oil and fat.

The **MQC** benchtop Nuclear Magnetic Resonance (NMR) analyser provides an alternative to extensive wet-chemistry methods. It is quick and easy to perform, simple to calibrate and requires minimal sample preparation. As such it is ideal for non-specialist laboratory personnel.



Method

Benchtop NMR detects the signal from oil and fat on a single filter sheet after the initial NMR signals from the solids and any remaining moisture have decayed completely. The remaining signal intensity correlates with the mass of fat, oil and grease (FOG) in the sample.

Calibration

Since NMR calibrations are always linear, only two well known standards are required to calibrate the analyser. However it is recommended that the instrument is calibrated using at least 6 standard samples with a known oil/fat content, evenly spread over the range of interest. The samples must be oven dried prior to the NMR measurement.

The calibration standards are prepared by adding 100 mg of the desired composition of fat, oil or grease (edible oil, animal fat, mineral oil, engine lubricant, etc.) to petroleum ether (100ml), followed by adding aliquots to clean filter sheets. After drying, the reference grease content (mg) of each standard is measured using a precision analytical balance to two decimal places
(± 0.01 mg).

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Measurements

To prepare a sample for analysis, a dried filter sheet containing filtration residuals is folded and placed into a small glass vial. The samples are then temperature conditioned at 40°C in a dry block for at least 20 minutes prior to the measurement by the **MQC**. The total measurement time for one sample is 5 minutes, excluding the time required for sample conditioning.

Results

Figure 1 shows a calibration obtained by measuring a set of calibration standards against the mass of grease/oil.

Figure 1. *NMR calibration generated for a set of standards prepared using the gravimetric data for the oil/fat mixtures. The correlation coefficient and standard deviation are 0.99995 and 0.62 mg respectively.*

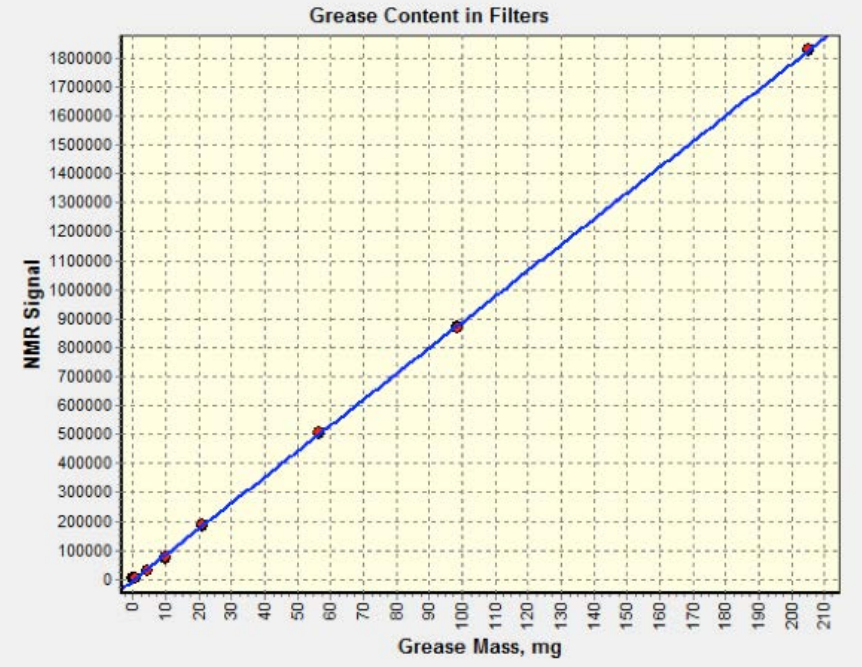


Table 1 shows that the NMR method is more accurate than Soxhlet extraction when compared against the reference grease content values determined by gravimetric analysis.

Table 2 shows the repeatability of the NMR method using one low and another more typical concentration.

Table 1. Soxhlet extraction and NMR results for grease content from the calibration standards

Sample ID	1	2	3	4	5	6	7	8	9
Reference grease content measured gravimetrically m_0, mg	0.6	0.8	0.8	4.5	10.0	21.3	59.4	98.4	205.0
Grease content determined by extraction method m_1, mg	0.3	0.6	3.4	5.3	8.4	20.1	54.8	94.5	193.3
Grease content measured by NMR method m_2, mg	0.9	0.9	1.0	3.7	8.9	21.2	56.9	97.7	205.4
Difference $\lvert m_0-m_1 \rvert$, mg	0.3	0.2	2.6	0.8	1.6	1.2	4.6	3.9	11.7
Difference $\lvert m_0-m_2 \rvert$, mg	0.3	0.1	0.2	0.8	1.1	0.1	2.5	0.7	0.4

Table 2. Results of NMR repeatability test

Grease Sample, mg	Repeat Measurements: Grease content by NMR, mg											Mean, mg	Standard deviation, mg
1.2	1.24	1.24	1.02	1.34	1.43	1.27	1.14	1.27	1.13	1.21	1.29	**1.23**	**0.11**
31.4	31.39	31.38	31.53	31.17	31.45	31.37	31.36	31.46	31.05	31.36	31.36	**31.35**	**0.13**

Conclusion

- The sensitivity of Oxford Instruments' **MQC** enables accurate measurements of grease /oil content on a single filter sheet.
- NMR measurement accuracy is better than the extraction method.
- Repeatability of the analysis measurements is good.
- The single sample measurement is rapid (typically 5 minutes).
- Minimal sample preparation is required, without the use of hazardous solvents and other media
- The NMR technique is non-destructive, so repeat measurements can be made conveniently.

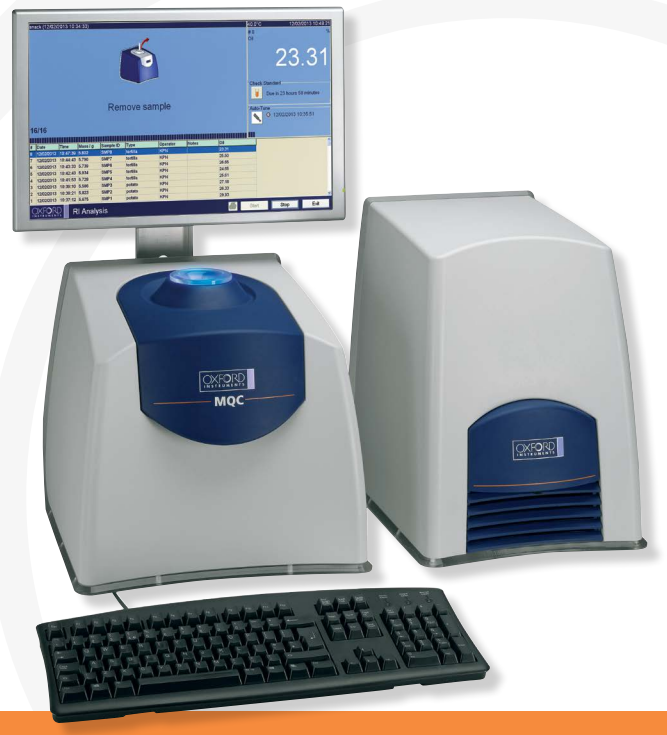
Complete Package

Oxford Instruments offers the following package tailored for accurate measurements of the grease content on filters:

- Oxford Instruments **MQC**23 NMR Analyser:
  - 0.55 Tesla (23.4 MHz) high homogeneity magnet
  - Probe for 23 mm diameter vials
  - Integrated system controller (no external PC required)
  - Integrated flat-screen display
- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** "Grease on Filters" application
- Test/tuning sample
- 23 mm diameter sample vials
- PTFE sample vial holders
- User manuals
- 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 (two decimal places, ±0.01 mg)



visit **www.oxford-instruments.com/MQC** for more information

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