Application Note AN M167

Oil in Water Analysis by Solid Phase Extraction

Keywords	Instrumentation and Software
Oil	ALPHA II FTIR spectrometer
Grease	OSS ClearShot™ Extractor
Wastewater	Transmission
Offshore	Macro
ASTM	Quant

About oil, grease and water pollution

Many international legislations acknowledge that the monitoring of oil and grease content of industrially used (waste) water is essential to protect aquatic and human life.

The 1974 US Clean Water Act mentions oil and grease as a conventional polluant. This term describes a water pollutant that is amenable to treatment by a municipal sewage treatment plant.

Wherever oil and grease are handled industrially and on a larger scale (e.g. offshore oil-rigs) their discharge into the environment should be tested.

Analysis of oil and grease in water

The current standard methods use highly flammable solvents such as pentane or hexane, to extract oil and grease from water. The extract is then analyzed by gas-chromatography (GC), or evaporated to measure the residue.

Since pentane and hexane are highly flammable and form explosive gas-air mixtures, the use of these solvents on oil platforms is not permitted.

Hence, the wastewater samples are transferred to a mainland lab, which is a very costly and time consuming procedure.

FTIR as ASTM approved alternative

The ASTM approved "Standard test method for solvent-free membrane recoverable oil and grease by Infrared determination D7575" is a more convenient alternative.

Briefly, this method quantifies oil and grease by processing a water sample through an oil capturing membrane and analyzing the membrane by transmission infrared spectroscopy. The membrane material does not block in the infrared range of interest.

Since this method does not use flammable and hazardous solvents, it can be performed on-site without moving the sample to the main analytical facility. This considerably reduces time for the analysis and enables near-real-time feedback and control for customers.

Fig. 1: OSS ClearShot™ oil and grease extractor.

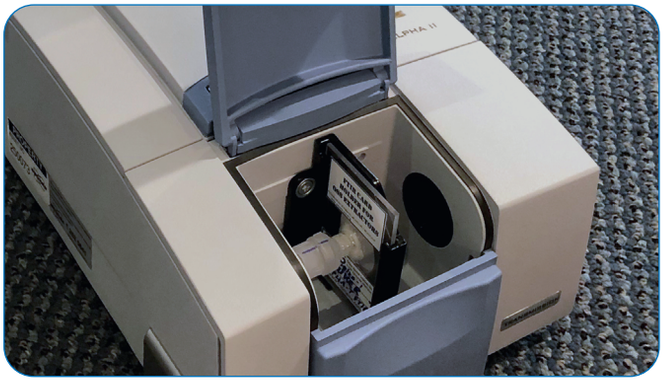


Fig. 2: Oil membrane mountend inside ALPHA II FTIR spectrometer.

What is needed: the analytical setup

FTIR Instrumentation

To perform the IR analysis, an ALPHA II FTIR spectrometer is used. The instrument has been designed to be operated by inexperienced personnel and provides high user guidance. An optionally integrated touch panel PC allows easy transport of the ALPHA II.

Oil and Grease Extractor

The ClearShot™ Orono Spectral Solutions, OSS, oil and grease extractor system is shown in Figure 1.
It consists of oil capturing membrane in a housing that is attached to a 2x3" plate. The plate fits to a standard transmission holder of any Bruker FTIR spectrometer.

Calibration and Software

The ALPHA II and OSS kit come with a certified calibration set. This also includes a calibration model and optimal measurement parameters, which are automatically built into user-friendly macros that perform the analysis.

The ASTM D7575 method uses solid-state calibration standards with known amounts of oil on the filter membrane. Thus, no calibration samples are needed to be prepared by the user.

How does it work: the ASTM D7575 procedure

Sample Preparation

The test method is very simple. It consists of drawing a homogenous water sample into a syringe and processing the sample through an extractor and drying the extractor using compressed air. The entire procedure takes only a few minutes per sample.

Measurement

The dried ClearShot™ extractor is simply placed into the transmission holder of Alpha II spectrometer and a spectrum is measured using the same spectroscopic settings used during the calibration procedure. A macro automatically guides the user through the method and calculates the amount of oil and grease according to ASTM D7575-11 method.

Results

Total analysis time including sampling and measurement is less than 15 minutes for most samples. Parallel processing allows for processing many samples simultaneously.
An example analysis report is shown in Fig. 4.

Quantification basics

Quantification is based on the measurement of transmission spectra of oil and grease captured by the membrane.

Oil and grease exhibit a distinct absorption band near 2920 cm^{-1} (Fig. 3). The intensity of this band is proportional to the concentration of analyte. Figure 3 shows spectra measured at different oil concentrations as well as the calibration method.

The detection limit is 1 ppm for a 10 ml sample using a 25 mm ClearShot ™ extractor. Lower/higher detection limits can easily be achieved using more/less volume and/or different sized OSS extractors).

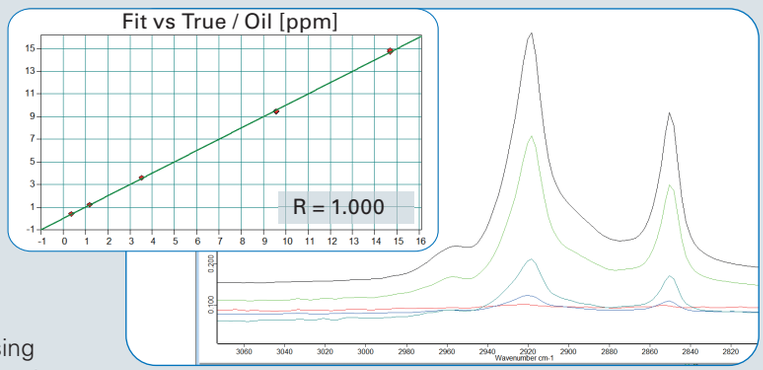
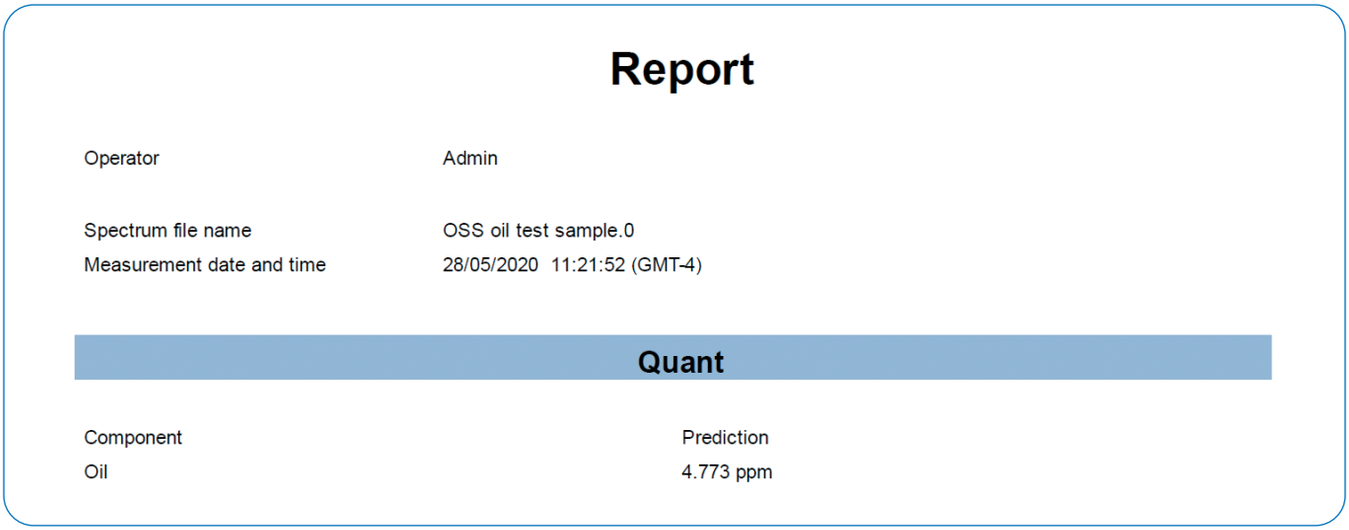


Fig. 3: Spectra of oil used for calibration. Oil concentrations from the bottom to the top spectrum are: 0.38, 1.18, 3.51, 9.56, and 14.69 ppm. Intensities are proportional to the oil concentration.



Report

Operator	Admin
Spectrum file name	OSS oil test sample.0
Measurement date and time	28/05/2020 11:21:52 (GMT-4)

Quant

Component	Prediction
Oil	4.773 ppm

Fig. 4: Example of analysis report.

Tracking the source of oil contamination

The use of FTIR technology offers additional functional benefits, including identifying the oil type in the sample. Since oil contamination can originate from different sources (engines, pumps etc.) tracing its origin is crucial. FTIR spectroscopy clearly identifies the type of oil, since spectra of different oils show very distinctive features.

The identification process is automatically performed by the system, without any additional effort or time. Oil from contaminated water is extracted using an OSS membrane, the oil spectrum is recorded, and then searched against a previously created reference library, that contains oils used in the concerned equipment in use.

Analysis will be completed automatically via the macro and the final report will include both oil concentration and identification.

Conclusions and summary

Bruker's ALPHA II FTIR in combination with OSS extractor allows fast, safe, accurate, precise and cost-effective quantification of oil and grease in wastewater at ppm levels in a few minutes. No hazardous chemicals are used in the analysis, no need for chemical explosion fume hoods, no additional glassware nor sensitive weigh balances.

In addition, FTIR spectroscopy enables tracing the origin of the contamination, hence providing crucial and timely information to stop the contamination from becoming larger or from happening again.

Bruker Optics Inc.
Billerica, MA · USA
Phone +1 (978) 439-9899
Fax +1 (978) 663-9177
info.bopt.us@bruker.com

Bruker Optik GmbH
Ettlingen · Germany
Phone +49 (7243) 504-2000
Fax +49 (7243) 504-2050
info.bopt.de@bruker.com

Bruker Shanghai Ltd.
Shanghai · China
Phone +86 21 51720-890
Fax +86 21 51720-899
info.bopt.cn@bruker.com

www.bruker.com/optics