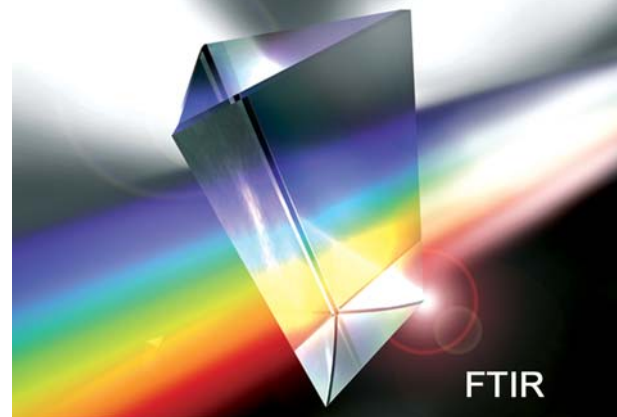


Application Note

Oil in water analysis under different European norms

Determination of hydrocarbons using FTIR-Spectroscopy



Depending on national norms the analysis of oil in water especial drinking water or ground are regulated by empirical solutions.

All norms have the intention to analyse the contents of hydrocarbons in water or ground using halogenated solvents like Trichloro-Trifluoro- Ethane (TTE) or Tetrachlormethane (CCl₄) for extraction.

Din 38409 H 18	Germany
BlueBook	United Kingdom
Blue Book	Sweden
NEN 6673/6675	Netherlands
ÖNORM M6608	Austria
CSN 83 0550	Slowakia
ISO/TR 11046	ISO Norm
T90-114	France
SFS 3010	Finland

Table 1: selection of regulations used for oil in water analysis in different European countries.

Why this method is used?

Just this method can fetch the hydrocarbons which are from high evaporation grade. So target is to get a sum parameter of all volatiles in form of the total contents of hydrocarbons. This style is not possible with the alternative GC method which will do a fine and exact analysis of all extracted hydrocarbons which are from lower evaporation grade. The FTIR method is also free from sample destruction which can be one effect from the GC analysis nature. So, at all FTIR is analysing another aspect of hydrocarbons than in comparison to the well known GC method. Unfortunately the halogenated solvents are not more allowed to be produced or are banned in some countries.

Nevertheless some laboratories got

- permission to do this analysis in their country,
- the method is still in a status to be allowed until the solvent is not more available
- other integrated the test as additional check to the GC method as house internal quality control.

Sample preparation

A defined amount of ground or water is treated with solvent to extract the hydrocarbons. The organic phase is separated from the water or ground, and send over a drying medium to remove the traces of remaining water amounts. The eluant will be filled into a quartz cell and measured in FTIR-Spectrometer.

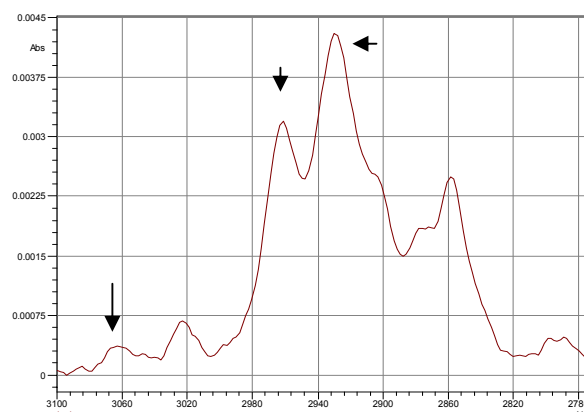


Fig.1: IR spectrum showing Oil generated by using CSN 83 0550 Norm, analytical wavelengths of interest are 2930, 2961 and 3061cm⁻¹ (marked with arrows)

Theory

Three analytical wavelengths from the FTIR spectrum are used to calculate the contents of hydrocarbons (Fig. 1). Depending on the source of the hydrocarbons aromatic and aliphatic elements can be inside of the extraction. Figure 2 represents diesel oil

which has no aromatic signal and figure 1 shows an oil sample with aromatic content.

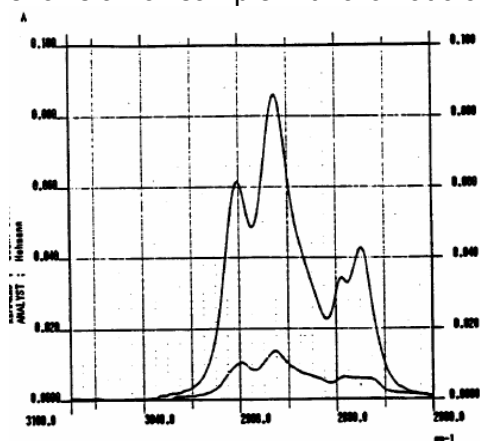


Fig. 2: IR spectra of two different concentration of diesel oil in extract in a 1 cm quartz cell, no aromatic signal at 3030 cm^{-1}

The oil will have signals at 3030 (aromatic), 2958 and 2924 (aliphatic) cm^{-1} for example when TTE is used. Only in Norm T90-114 four analytical wavelengths are used (Table 2). The detection limit for this analysis is very low and can be found with 5ppm for theoretical sample in a 5 cm cell in TTE.

The Norms

Differences in the norms are related to the solvent which is used for the analysis.

Some norms require a fresh fixing of factors or a calibration before the analysis using organic stock solution prepared from n-hexane, iso-octane and benzene. Another selection is a system based on hexadecane, pistane and toluene. These solvents are only examples. Best is to study each norm for the individual requirements

Based on the used solvent the analytical wavelengths will change.

Other norms use empirical equations with factors which are fixed like DIN H 18.

The concentration will be calculated with the absorbance values at analytical wavelength or substance maxima.

Norm	Solvent	Wavelength [cm^{-1}]
Din 38409 H 18	1,1,2-Trichloro-Trifluoroethane	2924, 2958, 3030
BlueBook UK	CCl_4 or 1,1,2-Trichloro-Trifluoroethane	2930, 2960, 3030
Blue Book S	1,1,2-Trichloro-Trifluoroethane	2930, 2960, 3030
NEN 6673/6675	CCl_4 or 1,1,2-Trichloro-Trifluoroethane	2925, 2958, 3030
ÖNORM M6608/S2120	1,1,2-Trichloro-Trifluoroethane	2924, 2958, 3030
CSN 83 0550	CCL_4	2930, 2961, 3061
ISO/TR 11046	1,1,2-Trichloro-Trifluoroethane	2925, 2958, 3030
T90-114	CCl_4	3290, 3380, 3420, 3510
SFS 3010	CCl_4	2925, 2930, 2960

Table 2: Listing of norms with analytical wavelengths and related solvents.

Instrumentation:

- FTIR-8400S or IRPrestige-21
- Cell holder for 1 up to 5cm cells
- 1 up to 5 cm quartz cells

Automatisation:

- IRsolution software
- Oil in Water Macro fixed to individual Norm
- Data acquisition and print manager, post run possible