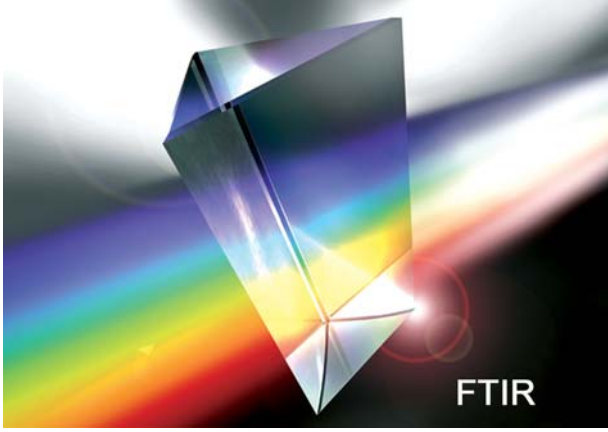
Application Note

Infrared Spectroscopy of FAME in Biodiesel following DIN 14078



Quantification of Ester –FAME- in Biodiesel Blends

European agriculture produces a lot of plants containing oil which will be used for commercial purposes. For the food market the rape seed oil is used as salad oil or as main part of margarine.
Actual the rapeseed oil is also a discussion point for the biodiesel aspects.
The quality of such biofuel and the blends of it are controlled by the Biodiesel regulation. One aspect of the control is the FAME (Fatty acid methyl ester) contents.

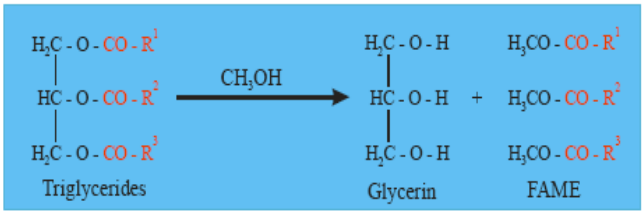


Fig. 1: Transesterification of tri-glycerides to FAME

Biodiesel like rape seed oil is rich on fat molecules which are treated in a transesterification process to contain a variety of esters. The tranesterification is necessary because motors cannot run with the natural rape seed oil which is too viscose.
In the figure1 the chemical treatment is shown in a short graph. The huge molecule of a tri-glycerol is cracked into the glycerine molecule and diverse FAME.

The blends of the biofuel are important to be controlled. This is possible with infrared spectroscopy. The reason is the simple identification of the mixtures. In the figure 2 the infrared spectrum of a mineral oil and a biodiesel are presented. The most important signal the ester related carbonyl bond at approx. 1745cm^{-1} is quantified in the manner to classify the blend of mineral oil and biodiesel. This part is regulated by the DIN EN 14078 norm.

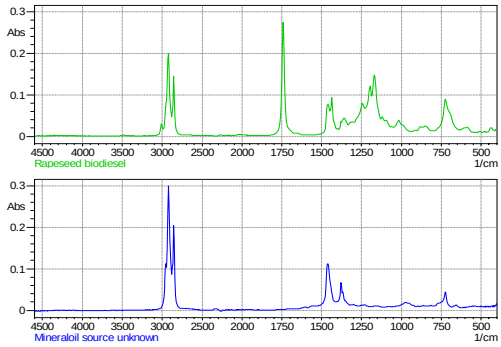


Fig. 2: Comparsion of Biodiesel and mineral oil – significant difference is to see in the ester signal at 1750cm^{-1}

Content of DIN EN 14078

The ester -CO signal is target of DIN EN 14078 from March 2004 and has the title "Liquid petroleum products -Determination of fatty acid methyl esters (FAME) in middle distillates- infrared spectroscopy method"

- The norm will focus on the measurement range of 1670 to 1820cm^{-1} and the signal of interest is at about 1745cm^{-1}.
- A cell with a thickness of 0.5mm and windows prepared from KBr, NaCl or CaF_2 should be used.
- For dilution and cleaning the solvent cyclohexane is to use.
- A calibration is to establish with 1,2,4,6 and 10g/l of FAME in cyclohexane

Sample preparation

In this application a KBr flow cell from Specac was used. The sample thickness was -as defined from DIN EN 14078- 0.5 mm in

length. As recommended from the norm 6 standards were prepared and measured. The measurement was done in the way that cyclohexane was used as reference and against this reference the sample was measured. For analysis only the part of the carbonyl bond was used for the calibration (Fig. 3).

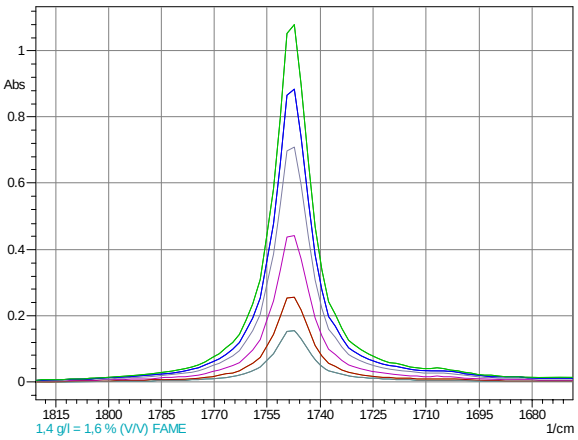


Fig. 3: Infrared spectrum of ester signal of 6 standard blends in the range of 1 to 11%V/V

The correlation coefficient is calculated to r2=0.999954. The result fulfilled the norm which requested a value higher 0.99.
One sample was checked against the calibration. The sample was declared with 5.26%V/V and the calibration found 5.37%V/V.

Table 1: Standard table

No.	Weight [g/L]	Volume [%V/V]	Unit [Abs]
1	1.4	1.6	0.154
2	2.3	2.6	0.256
3	3.96	4.5	0.439
4	6.4	7.3	0.702
5	7.9	9.0	0.876
6	9.7	11	1.071

Density of FAME d=880 kg/m^3

Procedure:
The solutions prepared from cyclohexane were transferred into the sample cell (3 times). Each time before sample was used the solvent was send through the system for rinsing and cleaning (3 times). The control of the baseline showed that no contamination of the cell was visible.

Instrumentation:
IRPrestige-21 with IRsolution Software
Cell Specac, KBr-window, 0.5mm pathlength
Sample preparation time: 5min incl. rinse times
Measurement technique: Transmission