SHIMADZU

Application News

Spectroscopy - FTIR

No. SCA-110-017

Iodine number determination using FTIR – ATR – Spectroscopy

The IR spectroscopy is a well-known technique in the field of foods analysis. Regards the wellness of the human an important aspect is the composition from oils and fats. High cholesterol values in blood are responsible for heart- and vessel disease. For this reason it is most important for producer and finisher of fats and oils to know the precise content of saturated and unsaturated fatty acids. From interest is also the valuable substance in terms of nutritional physiology which is reflected by the cis/trans portion of the unsaturated fatty acid. Special unsaturated fatty acids are essential like the linoleic acid and the arachidonic acid.
The complete chemical analysis should cover qualitative and quantitative determination of each fatty acid ester. Such analysis is relative complex and time consuming. The industry decided to use a number to identify the fats. The ester determination is dominated by the following numbers: acid, saponification, ester and iodine number.

The iodine number is by definition the amount of iodine in gram necessary to bound 100 g fat under decoloring. The iodine number is a degree for the content of unsaturated fatty acids. These will pick up elemental iodine by cracking the double bonding. The hardening is the reduction of double bonding for example whale oil has an iodine number of 120-140 and this will decrease to 12-70.

Since the IR spectrum shows precisely the cis and trans fatty acids it suggests itself as methods for the quantitative analysis (see. fig.1). The characteristic peaks from the cis unsaturated fatty acid at 3010 cm^{-1} and the trans unsaturated at 965 cm^{-1} easily (see fig.2 and 3).
Absorbance of water is visible in the range of 3400 and 1640 cm-1 as broad bands.

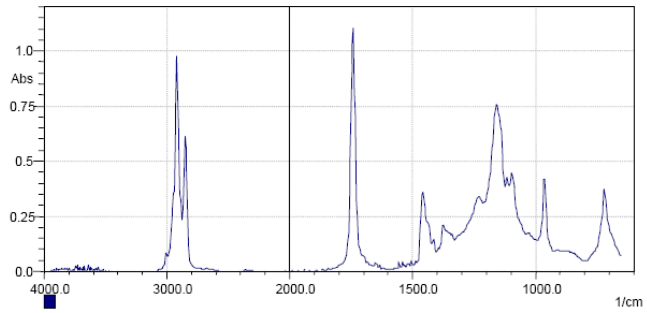

Fig. 1: Example IR spectrum of linseed oil made from linseed (flaxseed) measure with ATR accessory

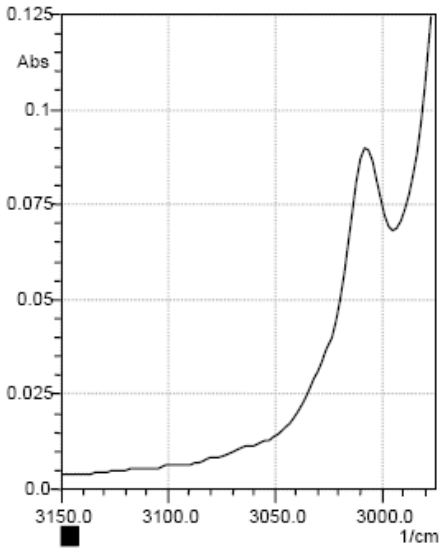

Fig. 2: IR spectrum from CIS unsaturated fatty acid, shown is range around peak 3010 cm^{-1}

With the significant peaks of water and the cis and trans unsaturated fatty acids are quantification procedures possible. In the following an example was calculated with 14 references. In a short overview the cis / trans values are shown (see fig. 4)

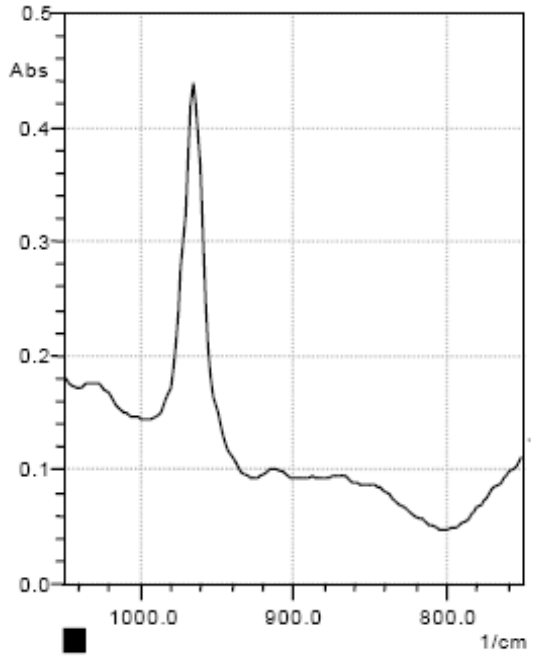

Fig. 3 IR spectrum from trans unsaturated fatty acid, shown is range around the peak at 960 cm^{-1}

File	Cis	Trans
JZ1.IRS	78.500	8.400
JZ2.IRS	77.000	8.600
JZ3.IRS	26.100	58.600
JZ4.IRS	29.100	55.600
JZ5.IRS	28.300	55.900
JZ6.IRS	134.100	0.000
JZ7.IRS	123.400	5.800
JZ8T.IRS	112.500	11.700
JZ9.IRS	80.200	29.200
JZ10.IRS	62.200	39.000
JZ11.IRS	13.400	0.000
JZ12.IRS	26.800	0.000
JZ13.IRS	5.800	11.100
JZ14.IRS	14.600	27.800

Fig. 4 Table of 14 standards and their cis and trans value

	Iodine number
JZ1.IRS	86.900
JZ2.IRS	85.600
JZ3.IRS	84.700
JZ4.IRS	84.700
JZ5.IRS	84.200
JZ6.IRS	134.100
JZ7.IRS	129.200
JZ8T.IRS	124.200
JZ9.IRS	109.400
JZ10.IRS	101.200
JZ11.IRS	13.400
JZ12.IRS	26.800
JZ13.IRS	16.900
JZ14.IRS	42.400

Fig. 5. Iodine number result after calibration of the references, correlation coefficient 0.99996, SEP 8.817E-003

The result from a PLS2 is shown in fig. 5. The calculation used two ranges dedicated to the cis/trans value for the calculation. The standard error of prediction (SEP) shows a good value for the modelling, and the correlation coefficient underlines this.

■ Conclusion

Conclusion is that this measurement procedure in combination with minimal sample preparation allows the quantitative analysis from iodine number from oils and fats.

■ Instrumentation

FTIR: IRPrestige-21
Software: PLS module
Accessory: DuraSampler II with KRS-5 Element and Diamond Prism