

Key Words

- Edible oil refining
- Iodine value
- IV
- Melting point
- Percentage solids
- Triglycerides
- FTIR
- Spectroscopy
- NIR

Introduction

Traditional measurements of iodine value (IV) for triglycerides is a laborious chemical assay requiring a laboratory to perform. It is slow, requires a sample to be removed from the process line and requires specialist equipment and skills. The time from needing to make a measurement to obtaining a result can be up to several hours, making iodine value measurements a case of "what did happen?" rather than "what is happening right now?".

Spectrometers allow continuous and detailed measurement of chemical concentrations in real time. Unfortunately the majority of process spectrometers are based on near infrared light, which is fundamentally less informative than mid infrared light. Conventional mid infrared spectrometers (which often use a Fourier transform are referred to as "FTIR spectrometers") have sensitive moving parts and fragile fibre probes - making them wholly unsuitable for production environments such as oil refineries.

The IRmadillo is a process analyser built using FTIR spectroscopy, but with static optics: removing the need for moving mirrors or fibre probes, and dramatically improving stability, reliability, and ruggedness.

Example use cases

This application note presents data from laboratory sampling of various different triglycerides with known IV measurements, to establish the performance of the IRmadillo. The expected use cases for this work range from control of reduction and hydrogenation processes through to melting point measurement and fractionation control.

All measurements were recorded at 90°C in a heated sample cell to best replicate process conditions.

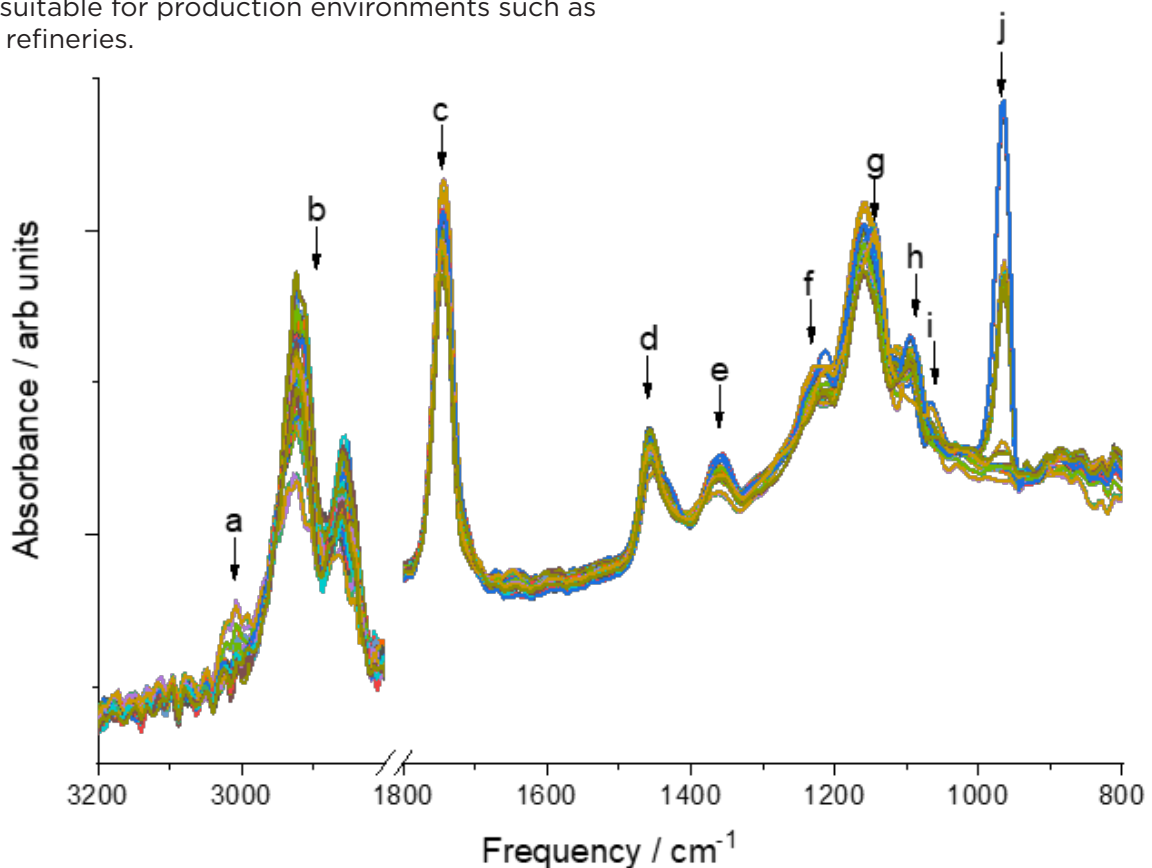


Figure 1: Spectra generated by the IRmadilloDiamond analysis of multiple triglycerides generated to produce a calibration model for iodine value and overlain to clearly show consistency and the key peaks of interest.

Results

The spectra of the different triglycerides are seen in Figure 1. It is possible to completely identify the different peaks seen with FTIR (not possible with NIR), where the band assignments are:

- a) C=C-H stretch
- b) C-H stretches
- c) C=O stretch
- d) CH₂ "scissors" vibration
- e) CH₃ bending
- f-i) C-O-C and C-C stretches
- j) C=C-H bend in trans bonds

A partial least squares (PLS) model can also be built to accurately measure the IV of the oils, and the calibration curve is shown below in Figure 2.

The accuracy for a full calibration ranging from 0 to > 250 IV is 7 g I₂ / 100 g oil. This value can be reduced by building smaller, more specialised calibrations - and it is possible to run multiple calibrations simultaneously on an IRmadillo if different process streams are treated through a single pipe.

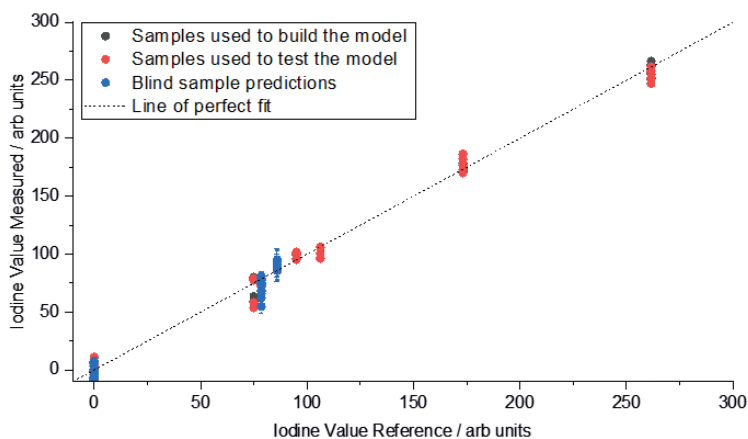


Figure 2: Measurement output of an IRmadillo installed in water washed oil inlet

Keep in Mind

The IRmadillo can be calibrated to measure a very large range of chemicals, all in real time. It has effectively measured phosphorus (P) free fatty acids (FFA), water/moisture and many different metals (Fe, Cu, Ca and Mg) simultaneously in a range of different oil types. Once calibrated, the IRmadillo becomes a universal chemical concentration meter - for all types of fats and oil processing.

Conclusions

This works shows that the IRmadillo FTIR spectrometer can measure different oils and oil-types and identify the different chemical features that define them. By building a chemometric calibration it can also be converted into an analyzer for the quantification of iodine value - in real time.

Next steps are for an on-line installation directly into an oil processing facility to demonstrate this in the real world. Keit already has installations in oil refineries measuring FFA and P, and are expanding into IV



Hazardous area installations

Keit recognises that the many of the applications requiring IV measurement - such as hydrogenation - will take place in hazardous areas and at elevated temperatures and pressures.

The IRmadilloEx model was designed specifically for these environments, and can operate up to 220°C and 600 psig.

Visit www.keit.co.uk for more information on the IRmadillo's certification and specifications.

Contact us



+44 (0) 1235 431260



(Sales) enquiries@keit.co.uk



(Product Support) support@keit.co.uk



www.keit.co.uk



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