The high-speed camera HPV-1 Hyper Vision enables views into a previously unknown world

IMPRINT

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Flower Power

GC for quality control of Biodiesel

Rising fossil fuel costs have contributed to making renewable energy sources an attractive option in the present energy situation. The potential of renewable raw materials has already been widely acknowledged and promoted in the European Union. As an admixture to diesel fuel or petrol, renewable sources aid in prolonging the limited availability of fossil-based energy sources. The rapeseed plant is one of several suitable biodiesel raw materials and its robustness allows cultivation all over Europe.

Untreated rapeseed oil is very viscous and must be preheated prior to combustion in a diesel engine. The engine technology required for this purpose is cost-effective only for large diesel aggregates used in ships or heavy lorries. For use in automobiles and smaller trucks, chemically altered rapeseed oil is at present more economical. This alteration is achieved via transesterification with methanol, whereby glycerin esters present in the plant oil are converted into fatty acid methyl esters (FAME, Fatty Acid Methyl Esters, see Figure 1). These remain fluid even at sub-zero temperatures and can be used instead of diesel in many diesel-powered vehicles.

Growing need for biodiesel in Europe

The most important market potential for biodiesel is its application as an admixture to fossil-based diesel fuels.

Promoted via tax subsidies, the growing demand from large oil companies is leading to a continuous growth of biodiesel production in Europe. As the biodiesel production process is not only limited to rapeseed oil but can be applied to any plant-derived oil as well as animal fats, standardized quality control is essential. This has been documented in the DIN EN 14214 Standard which also takes into account that biodiesel as a natural product is subject to a limited shelf life. DIN EN 14214 combines all analytical procedures – for the most part chromatographic and spectroscopic measurements – and specifies under which conditions these methods are to be applied for biodiesel quality control. Shimadzu offers a complete solution for these analytical procedures.

Biodiesel quality control requires state-of-the-art technologies

Shimadzu has been involved from the beginning with biodiesel quality control according to DIN EN 14105, one of three gas chromatographic analytical methods. Testing for the completeness of transesterification of glycerin esters to fatty acid methyl esters via quantification of the remaining mono-, di- and triglycerides represents an important aspect of daily process control (Figure 2).

This method simultaneously determines the amount of free glycerin in biodiesel. The determination of excess methanol is also carried out via gas chromatography according to DIN EN 14110.

Analysis of glycerin esters according to DIN EN 14105 places high demands on the gas chromatograph and the capillary column, while separation of the mixture from glycerin up to the triglycerides requires operation at a GC oven temperature range from 60 °C up to 400 °C.

The method is based on the "on-column" injection technique whereby the sample is injected directly onto the GC column using a syringe. To accommodate the syringe needle, the column must have a minimum internal diameter (ID) of 0.53 mm. As the column ID has a direct effect on the signal resolution as well as retention times, small column IDs are normally preferred in gas chromatography. For "on-column" injection a retention gap is therefore applied. This is a 2 m long uncoated capillary with an ID of 0.53 mm coupled to the separation column inlet (Figure 3). The coupling of retention gap and main column has proven to be maintenance-intensive in biodiesel analysis. Due to the extended oven temperature range this connector eventually leaks and requires readjustments in order to guarantee gas-tightness of the system.

Shimadzu develops new technologies

Innovative technologies for sample preparation and analysis have contributed to Shimadzu's reputation as know-how leader in biodiesel analysis. This has been achieved in close cooperation with Shimadzu's customers in Europe, and biodiesel producers worldwide can now take advantage of these new technologies.

A specially developed liner for "simple on-column" injection offers the most up-to-date simplification of biodiesel analysis. In contrast to standard on-column injection, a glass liner is used instead of a retention gap (Figure 4). This technique features advantages in terms of simple installation as well as improved gas-tightness. The liners are deactivated and are available for all commercial fused silica capillary columns. Using the "simple on-column" liners for analyses according to DIN EN 14105 resulted in improved reproducibility of peak areas and retention times when compared with the retention gap.

The higher reliability and simple operation turns the "simple on-column" technique into an attractive alternative to the standard methods. Present trends towards achieving faster and better chromatographic results by using capillary columns with small internal diameters (fast GC) will further boost the use of this technique.

Shimadzu's GC-2010 system has already been optimized for fast and efficient analysis. The advantages of this system are higher sample throughput, faster results and answers in quality control and for process optimization as well as improved detection limits. The "simple on-column" injection technique greatly simplifies the use of small internal diameter columns in conjunction with on-column injection.

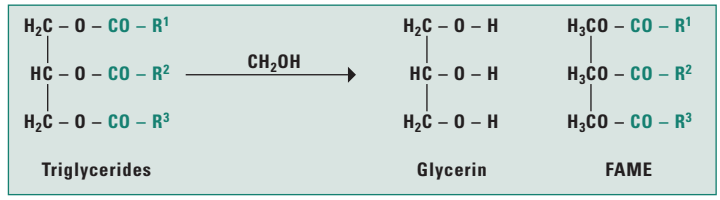

Figure 1: Transesterification of glycerin esters to fatty acid methyl esters (FAME). The notations R^1, R^2 and R^3 refer to the fatty acids (mainly palmitic acid, oleic acid, linoleic acid and stearic acid).

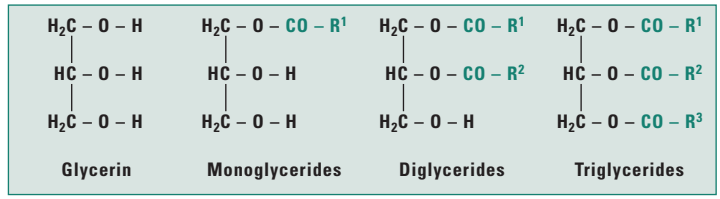

Figure 2: Quality control of biodiesel according to DIN EN 14105 consists of the quantitative determination of glycerin as well as glycerine ester content in biodiesel after transesterification

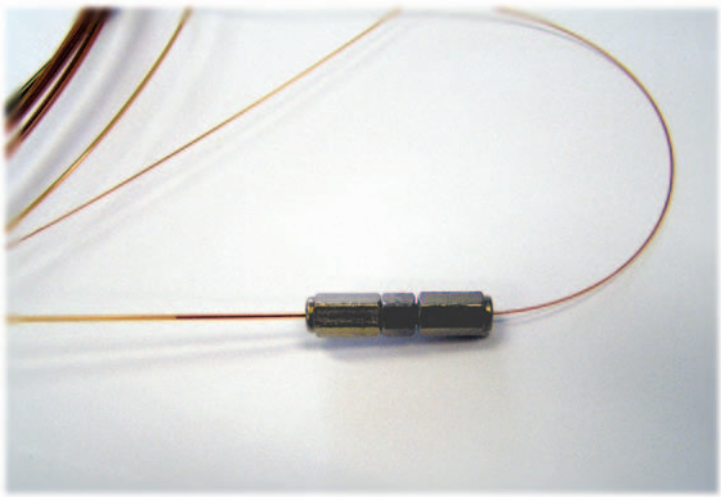

Figure 3: Coupling of the retention gap to the separation column

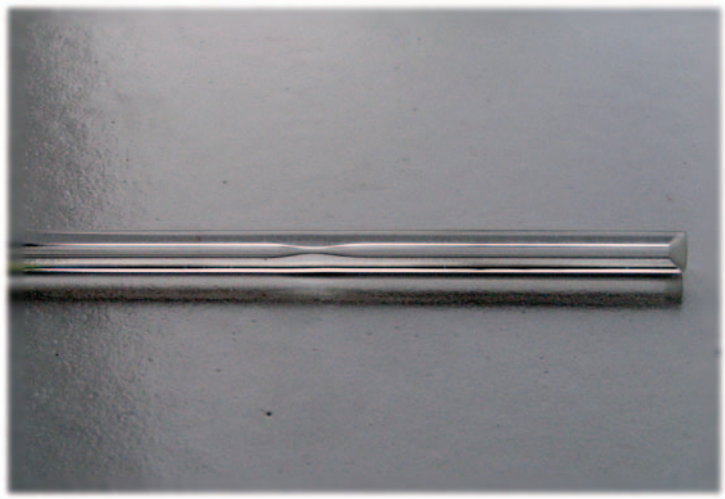

Figure 4: Shimadzu liner for "simple on-column" injection technology