

Analysis of Methanol in Biofuel by Headspace GC-FID

Method 1:

Instrumentation: Perkin Elmer Autosystem XL GC System ([Ctrl + Click to follow link](#))

In this application the biodiesel samples are analyzed by headspace-GC-FID.

The biodiesel samples are shaken and heated to 60 °C for a given time. Methanol is enriched in the gas-phase because of its low-boiling point, while the high-boiling point compounds remain in the liquid phase. When the equilibrium is reached, a volume is drawn from the gas phase(headspace) of the vial and injected into the GC system.

GC Column: Several types of GC columns are listed in the EN 14110 method as possibilities. Any column that delivers resolution and a symmetrical peak for methanol is acceptable. The columns included in this application note are:

- 30 m x 0.32 mm x 1.8 µm BAC-1 (PerkinElmer Part No. N9316579)
- 30 m x 0.32 mm x 3.0 µm Agilent Select Biodiesel for Methanol (Part No. CP9083)
- 30 m x 0.28 mm x 3.0 µm Elite-1 (PerkinElmer Part Nos. N9316025 or N9307067)

A calibration curve containing 0.01, 0.1 and 0.5 % (m/m) methanol in FAME, in accordance with DIN EN 14110 is prepared. 2-propanol is added as internal standard.

This calibration curve is shown in Figure 3, confirming the high linearity obtained with this system.

Also the reproducibility of measurements is calculated, as shown in Table 2. Reproducibility is higher than the requirements according to DIN EN 14110.

In conclusion this HS-GC-FID system, using a headspace autosampler, represents an efficient and precise solution for the methanol determination in biodiesel. A proper autosampler can prepare several samples simultaneously to assure the high sample throughput.

Instrument settings

HT200H	Headspace injection
Autosampler	Injection volume: 0.5ml Oven temperature: 60°C Shaking
GC	Injector temperature: 150°C Split ratio: 1:10 Oven program: 50°C isothermal Carrier gas: Helium 35cm/s
Columns	30m x 0.32mm x 1 µm
Detector	FID 150°C

Table 1: Instrument settings for methanol in biodiesel analysis

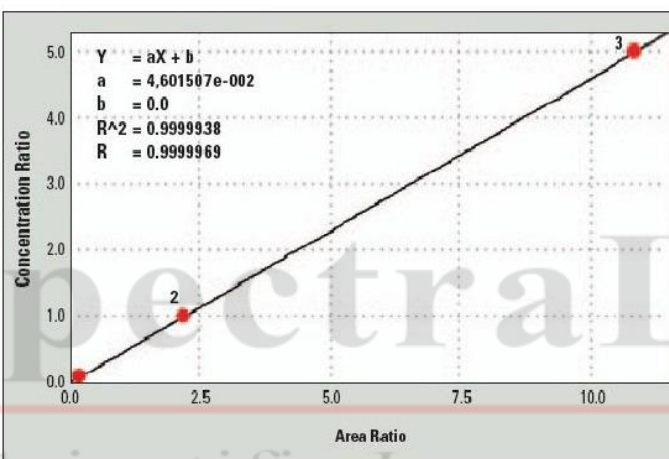
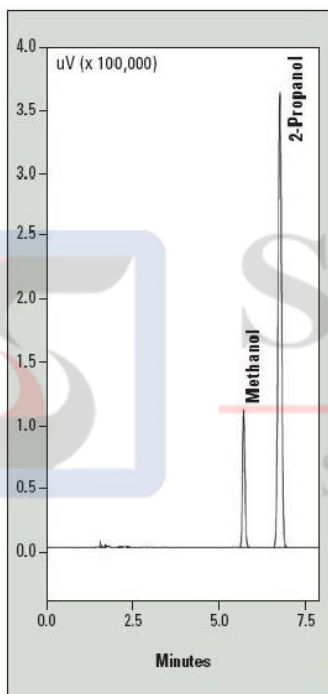


Figure 3: Linearity results (calibration curve of methanol in biodiesel)

Sample	1	2	3
Average concentration % (m/m)	0.009	0.100	0.500
Reproducibility standard deviation % (m/m)	0.00005	0.00100	0.00485
Reproducibility % RSD	0.54	0.99	0.97

Table 2: Reproducibility results

References:

- <http://www.hta-it.com/en/documentation/methanol-in-biodiesel.html>
<https://www.chem.agilent.com/Library/applications/5990-8984EN.pdf>
http://www.perkinelmer.ca/CMSResources/Images/44-74214APP_MethanolbyHS-GCinBiodiesel.pdf