

Application Note No. 073

The Automated On-Line Sampling and Analysis of Glycerol in Biodiesel

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Automated on-line sampling

- *Automated sample preparation*

Background

Biodiesel is a renewable fuel and can be made from most vegetable oils, for example oilseed rape and sunflower oil. It causes less pollution than fossil fuels and is mainly used in the transport industry. It is produced by trans-esterification of the oil, producing the oil methyl ester (biodiesel) and glycerol, which settles out. Through removal of the glycerol, biodiesel can be used in most diesel engines with little or no modification either on its own or as a mixture with conventional or low sulphur diesel. Here, a simple on-line method is presented as a way of analysing glycerol in biodiesel down to the necessary detection limit of 500 ppm.

Instrumentation

- ATAS Optic 2-200 programmable injector
- Focus Autosampling Robot
- HP5890 with FID
- SGE SolGel Wax 0.25 x 30 m x 0.25 mm i.d. column

Principles

- Take a sample of biodiesel from the stream using the Focus flow cell
- Add internal standards, the reagents ethanol and n-hexane and mix
- Inject 1 μ L in split mode

Chromatogram

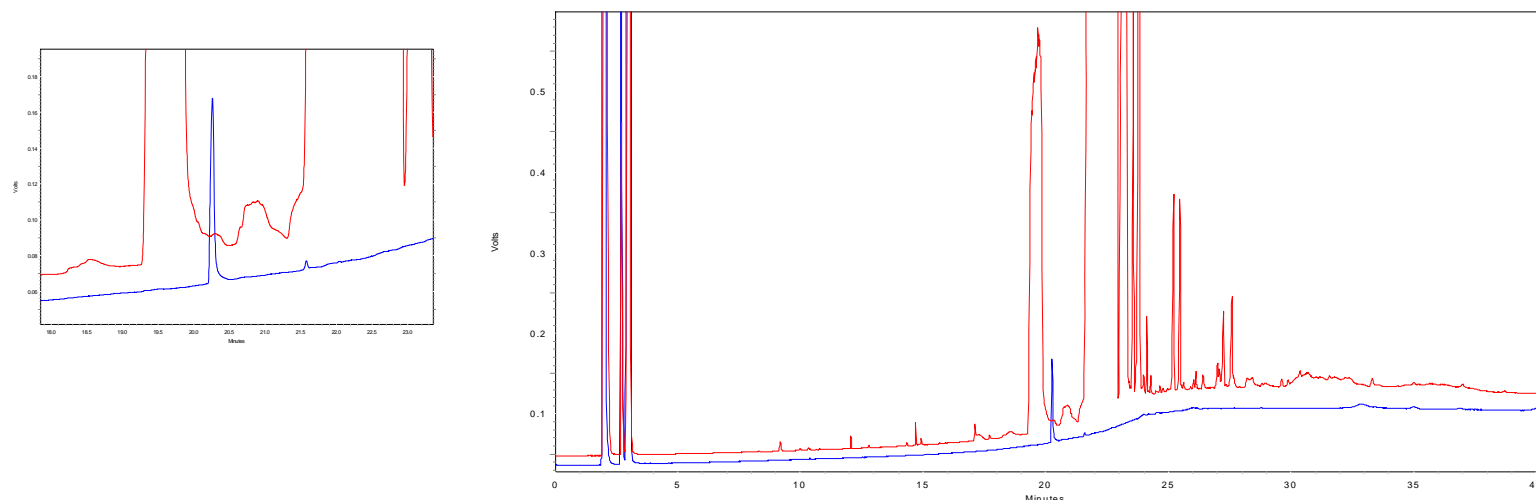


Figure: *Upper trace:* Injection of 1 μ L of Biodiesel; *Lower trace:* injection of 1 μ L of 500 ppm glycerol; *Insert:* Close-up of glycerol peak

We would like to thank Duncan Taylor from SGE Europe for providing the column for this application.

Optic Conditions:

- Liner: Fritted
- Mode: Split
- Injection volume: 1 μ L
- Gas Flows: Split: 10 ml/min
- Initial temperature: 60 °C
- Ramp rate: 4 °C/s
- Final temperature: 280 °C
- Final time: 20:00 m:s
- Transfer pressure: 14 psi
- Transfer time: 2:00 m:s
- Initial pressure: 14 psi
- Final pressure: 14 psi

GC conditions:

- Column: SGE SolGel Wax 0.25 30 m x 0.25 mm i.d.
- Initial Temperature: 50 °C (2 mins)
- Ramp: 10 °C/min to 280 °C (16 mins)
- FID temperature: 300 °C