

Determination of Free and Total Glycerine and mono-, di-, triglyceride content in B-100 Biodiesel Methyl Esters according to EN 14105

AN078v2; Nov 2023, SCION Instruments

Introduction

The European Standard method, EN-14105, is used to determine the free glycerol and residual mono-, di- and triglyceride contents in fatty acid methyl esters (FAME) typically intended for addition to mineral oils. Total glycerol content is calculated from the results obtained. The method is suitable for FAME from rapeseed, sunflower and soybean oils, but is not suitable for FAME produced from or containing coconut and palm kernel oils due of the problem of peak overlapping. This method and ASTM D6584 are two of the most used standardized analytical methods used for the analysis of biodiesel.

Experimental

The SCION Biodiesel analyser for EN 14105 is based on our 8300-GC platform, a cold-on-column injector, a SCION-Glycerides Inert Steel analytical column with a 2m retention gap and a High-Temp FID Detector. Analytical conditions for the EN 14105 analysis can be found in table 1.

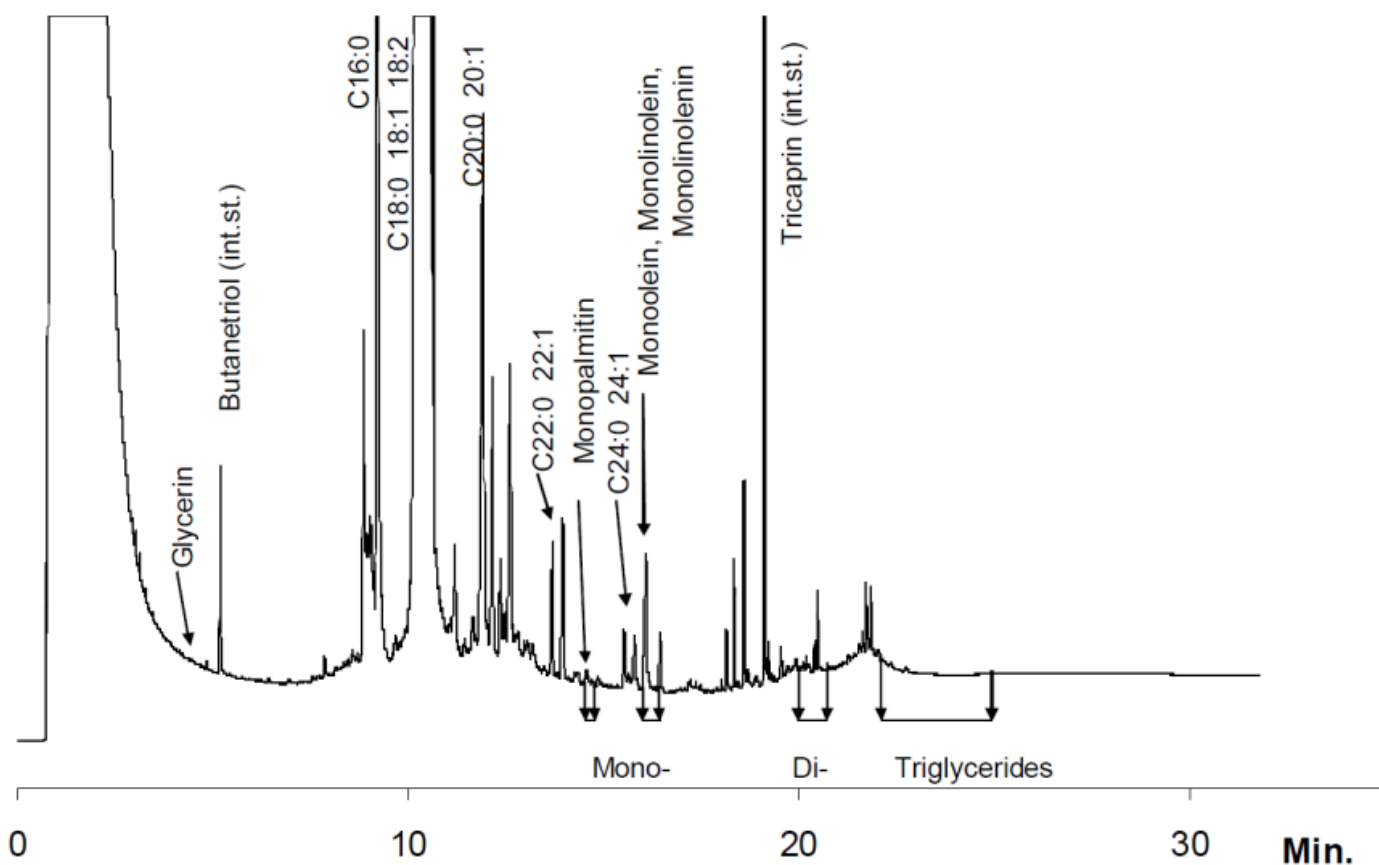


Figure 1. Biodiesel Chromatogram

APPLICATION NOTE

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Table 1 GC conditions

| Parameter | Settings |
|------------------|--|
| Injector | Cold-on-Column |
| Injection Volume | 1 μ l |
| Column | 10m x 0.32mm x 0.10 μ m SCION-Glycerides Inert Steel (SC38613) w/ Ret. Gap |
| Carrier Gas | Helium @ 80 kPa |
| Oven Program | Start @ 50°C (1 min) 15°C/min to 180°C 7°C/min to 230°C 10°C/min to 370°C End @ 370°C hold 10 min. |
| Detector | FID @ 380°C |
| Software | Compass CDS |

Sample Preparation

Calibration mixtures and internal standard solutions were prepared according to the method. 100 mg of homogenized sample was accurately weighed in a 10 mL vial. Then, using a glass syringe, the following was added to the vial: 10 μ L of internal standard No. 1 stock solution, 100 μ L of internal standard No. 2 stock solution and 100 μ L of MSTFA. Care was taken to ensure there was no contact with moisture. The vials were hermetically sealed and shaken vigorously. After storing the vials at room temperature for ~ 15 minutes, 8 mL of heptane was added to each. Then, 1 μ L of the reaction mixture was automatically injected into the gas chromatograph according to the instrumental conditions described below (see Table 2 for typical results).

Results

Calibration curves were obtained for glycerin, monoolein, diolein and triolein. Calibration curves for glycerin and monoolein, shown in Figures 2 and 3, are indicative of system performance for the application.

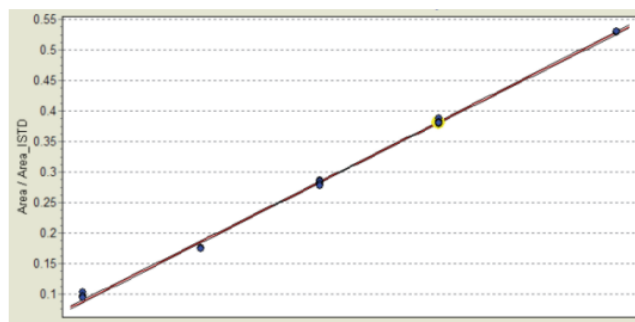


Figure 2 Biodiesel calibration 001 – Glycerin

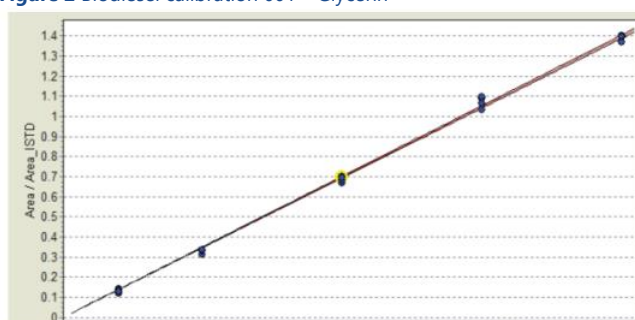


Figure 3 Biodiesel calibration 001 – Monoolein

Table 2 Repeatability results for B-100 biodiesel

| Compound | Average (% m/m) | %RSD |
|----------------|-----------------|------|
| Glycerin | 0.009883 | 1.5 |
| Monoglycerides | 0.687308 | 5.1 |
| Diglycerides | 0.0663714 | 2.4 |
| Triglycerides | 0.060268 | 3.6 |

Conclusion

This application note demonstrates the suitability of SCION Instruments Analyser for EN 14105, for the analysis of biodiesel. The calibration curves and repeatability data demonstrate good system integrity. Therefore, the system is well suited to the analysis of free and total glycerol and mono, di and triglyceride content in biodiesel in accordance with the standard method EN 14105

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| Ordering Information for the 8300 GC | |
|--|-------------|
| Part | Part Number |
| 8300-GC, with S/SL inlet and FID detector (120V) | 839001701 |
| 8300-GC with S/SL inlet and FID detector (230V) | 839001702 |
| 8400 PRO Autosampler for 8300 and 8500 GC | 840000001 |
| Suggested Consumables | |
| Part | Part Number |
| 15% Graphite/85% Vespel Ferrule 1/16" with 0.4 mm hole pk/10 | 41312148 |
| BTO Septa 9 mm, pk/50 | CR298713 |
| 10 µL fixed needle syringe, 5 cm, 0.47 mm OD, 26 g conical needle | 41312133 |
| Vial, 9-425 Screw Thread, 2 mL Clear Glass 12 x 32 mm Flat Base with Label, pk/100 | 41311000 |

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