



Biofuel Series

Laboratory Equipment

Report

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Definition of quality standards and equipment required for the measurement

Biodiesel is defined as the mono alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, for use in compression-ignition (diesel) engines. Before blending with diesel fuel biodiesel should match some specified quality standards. The quality standards required for pure biodiesel are gathered in a biodiesel specification. The required quality standards of biodiesel specification are listed in the table below.

| Specifications | Units | U.S. | EU |
|--------------------------------|--------------------|----------------|---------------|
| Test-Method | | ASTM D6751-07b | EN 14214:2003 |
| Density at 15°C | kg/m ³ | | 860 - 900 |
| Viscosity at 40°C | mm ² /s | | 3.50 - 5.01 |
| Flash point | Degree Celsius | 93 min | 125 min |
| Distillation, T90 | Degree Celsius | 360 max | |
| Carbon residue | % mass | 0.05 max. | - |
| Cetane number | | 47.0 min | 51.0 min |
| Sulphated ash content | % mass | 0.02 max | 0.02 max |
| Water & sediment | % vol | 0.05 max. | 500 max |
| Total contamination | % mass | - | 24 max |
| Copper band corrosion, 3h 50°C | rating | No.3 | Class-1 |
| Oxidation stability, 110°C | hours | 3min | 6.0 min |
| Acid value or number | mg KOH/g | 0.50 max | 0.50 max |
| Iodine value | | | 120 max |
| Ester content (C14-C24) | % mass | - | 96.5 min |
| Methanol content | % mass | 0.20 max | 0.20 max |
| Glycerides | | | |
| Monoglyceride | % mass | - | 0.80 max |
| Diglyceride | % mass | - | 0.20 max |
| Triglyceride | % mass | - | 0.20 max |
| Free Glycerol | % mass | 0.020 max | 0.020 max |
| Total Glycerol | % mass | 0.240 max | 0.250 max |
| Sulfur content | ppm | 15 max. | 10 max |
| Group I metals (Na+K) | ppm | 5 max | 5 max |
| Group II metals (Ca+Mg) | ppm | 5 max | 5 max |
| Phosphorus content | ppm | 10 max. | 10 max |

I Definition of quality standards

1 Density (EN ISO 3675, EN ISO 12185)

Biodiesel generally have higher densities than mineral diesel (EN 590 820-845 kg/m³ at 15°C). Density increases with a decrease in chain length and with unsaturation. This can impact on fuel consumption as fuel introduced into the combustion chamber is determined volumetrically.

2 Viscosity (EN ISO 3104, ISO 3105, D445)

Viscosities of neat vegetable oils are many times higher which leads to serious problems in unmodified engines. The increase in viscosity results in poor atomization and incomplete combustion which leads to coking of injector tips. This results in engine power loss. Biodiesel still has higher viscosity than mineral diesel (3.50-5.00 mm²/s at 40°C vs 2.00-4.50 mm²/s). Viscosity decreases with unsaturation but increases markedly with contamination by mono, di or triglycerides.

3 Flash Point (ISO 3679, IP 523, IP 524, D93)

Pure rapeseed methyl ester has a flash point value of up to 170°C. This method is therefore looking at residual components within the fuel that are combustible, especially methanol which is a particular hazard due to its invisible flame.

4 Distillation (T90 AET)

Biodiesel is distilled at atmospheric pressure. At least 90 % of biodiesel should be distilled with a maximum temperature of 360 °C.

5 Carbon Residue (EN ISO 10370)

The Carbon Residue is the material left after evaporation and pyrolysis of a sample fuel. This is a measure of the tendency to of a fuel to produce deposits on injector tips and the combustion chamber. For FAME samples it is an indication of the amount of glycerides, free fatty acids, soaps and catalyst residues remaining within the sample

6 Cetane Number (EN ISO 5165, D613)

This serves as a measure of ignition quality. This is the most pronounced change from vegetable oil to the trans-esterified product. Fuels with low cetane numbers show an increase in emissions due to incomplete combustion. Palm Oil and Tallow derived biodiesels have the best cetane numbers.

7 Sulphated Ash (ISO 3987, D874)

Ash describes the amount of inorganic contaminants, such as catalyst residues, remaining within the fuel. Ash is related to engine deposits on combustion.

8 Water Content (EN ISO 12937)

As FAME is hygroscopic it can pick up water in storage and as such there can be problems meeting the specification. At around 1500 ppm the solubility limit is reached and the water bottoms out. Free water promotes biological growth and the reverse reaction turning biodiesel to free fatty acids.

9 Total Contamination (EN 12662)

Total contamination is defined as the insoluble material retained after the filtration of a heated sample over a standardized 0.8m filter. FAME samples with high quantities of insoluble materials tend to cause fuel filter and injector blockages. High concentrations of soap stock are normally associated with high total contamination.

10 Copper Strip Corrosion (EN ISO 2160, D130)

This is defined as the likelihood to cause corrosion to copper, zinc and bronze parts of an engine. A polished metallic strip is heated at 50°C for 3 hours, washed and compared to standards. Corrosion is likely to be caused by free acids or sulfur compounds. However FAME gives consistently good results in this area and is unlikely to fail due to the low sulfur content.

11 Oxidation Stability (EN 14112)

The Oxidative stability specification is defined as a minimum Rancimat induction period of six hours. Essentially a fuel is heated at 110°C in a constant air stream and the formation of volatile organic acids is detected. This property relates to the overall storage stability of the fuel and the higher the degree of unsaturation (double bonds) within the FAME molecules gives a decrease in oxidative stability. This can be improved with the addition of antioxidant additives.

12 Acid Value (EN 14104, D664)

Acid value is a measure of mineral acids and free fatty acids contained in a fuel sample. It is expressed in mg KOH required to neutralize 1g of FAME. High fuel acidity is linked with corrosion and engine deposits.

13 Iodine Value (EN 14111)

Iodine number is a measure of total unsaturation (double bonds) within the FAME product. It is expressed as the grams Iodine required to react with 100g of FAME sample. High Iodine value is related to polymerization of fuels, leading to injector fouling. It is also linked to poor storage stability.

14 Ester Content (EN 14103)

This is measured using gas chromatography and is restricted to esters falling within the C14-C24 range. It is ultimately a test for reaction conversion. Linolenic and polyunsaturated esters are

controlled as they have been shown to display a disproportionately strong effect on oxidative stability.

15 Methanol Content (EN 14110)

Methanol can be removed from FAME by washing or distillation. High methanol contents pose safety risks due to the very low flash point of methanol.

16 Glycerides (EN 14105, EN 14106, D6584)

There is a limit on the mono, di, and triglycerides of no more than 0.80%, 0.20% and 0.20% respectively. Total glycerol is the sum of the bound and free glycerol and must not exceed 0.25%/ Failing to meet the spec implies low conversion to ester and deposit formation on injectors and valves.

17 Group I Metals

Sodium and Potassium are limited to a combined 5ppm. These arise from the addition of catalyst, and result in high ash levels in the engine.

18 Group II Metals

Calcium and Magnesium are limited to a combined 5ppm. These may arise from the addition of hard water in the washing process. Calcium and Magnesium soaps have been related to injector pump sticking.

19 Phosphorous Content (EN14107, D4951)

The phosphorous limit is approx 10 ppm and normally arises from phospholipids within the starting material or from addition of phosphoric acid in the production process. High phosphorus fuels are suspected of poisoning catalysts and increasing emissions.

20 Sulfur content (ASTM D 5453)

Limits: 0.05 % mass maximum

The measure is implemented by UV spectrophotometry

II Equipments for assessing biodiesel quality

1 Density (EN ISO 3675, EN ISO 12185)



Densimeter



Cylinder for measuring

2 Viscosity (EN ISO 3104, ISO 3105, D445)



Thermostated water bath



Viscosity tube

3 Flash Point (ISO 3679, IP 523, IP 524, D93)



Flash point apparatus

4 Distillation (T90 AET)



Distillation apparatus

5 Carbon Residue (EN ISO 10370), Sulphated Ash (ISO 3987, D874)



Muffle furnace

6 Cetane Number (EN ISO 5165, D613)



Cetane number analyzer

7 Water & sediment Content (EN ISO 12937)



Centrifuge with tubes

8 Total Contamination (EN 12662)



Filtration bank

9 Copper Strip Corrosion (EN ISO 2160, D130)



Copper strip corrosion apparatus



Copper strip corrosion chart

10 Oxidation Stability (EN 14112)



Rancimat

11 Acid Value (EN 14104, D664), Iodine Value (EN 14111)



Titration apparatus

12 Ester Content (EN 14103), Methanol Content (EN 14110), Glycerides (EN 14105, EN 14106, D6584)



Gas chromatograph analyzer

13 Group I Metals, Group II Metals, Phosphorous Content (EN14107, D4951)



ICP analyzer

14 Sulfur content (ASTM D 5453)



UV-visible spectrophotometer