Application Note AN-R-009

Oxidation stability of fatty acid methyl esters (FAME, biodiesel)

Reliable and accurate determination of the oxidation stability of biodiesel according to EN 15751

Biodiesel, also known as fatty acid methyl esters (FAME), is used just like petroleum-based diesel. Fatty acid methyl ester biodiesel can also be blended with petroleum diesel fuel in any ratio for use in diesel engines [1]. Biodiesel fuel produces fewer emissions, is sustainable, biodegradable, and environmentally friendly, and has good lubricating properties. Transesterification of glycerides in vegetable oils, animal fats, or organic waste with monohydric alcohols (e.g., methanol or ethanol) can make FAME.

Antioxidants, whether natural or added (e.g., ascorbyl palmitate), inhibit FAME autoxidation and help to prolong shelf life. Both the quality and antioxidant capacity of biodiesel need to be monitored. One of the most important parameters to measure is oxidation stability. The 893 Professional Biodiesel Rancimat determines the oxidation stability of biodiesel according to EN 14112, EN 15751, and EN 16568 standards.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on biodiesel with and without added antioxidants (Table 1).
For measurements of biodiesel with antioxidants, 10 mg of ascorbyl palmitate was added to 100 mL of biodiesel.

EXPERIMENTAL

The determinations are carried out using an 893 Professional Biodiesel Rancimat (Figure 1).
An appropriate amount of sample is weighed into the reaction vessel, and then the analysis is started.
The biodiesel sample is exposed to an airflow at a constant temperature of 80–150 °C using the biodiesel Rancimat method. In this way, highly volatile secondary oxidation products are transferred along with the airflow into the measuring vessel where they are absorbed in the measuring solution.

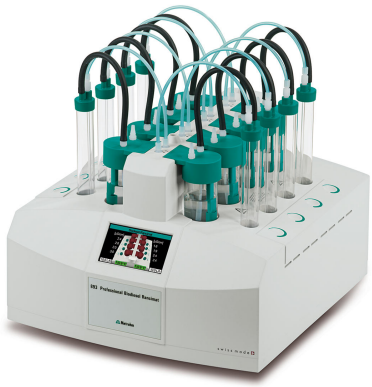

Figure 1. 893 Professional Biodiesel Rancimat equipped with measuring and reaction vessels for the determination of oxidation stability of biodiesel.

The conductivity of the measuring solution is registered continuously. The formation of secondary oxidation products leads to increased conductivity of the solution. A good indicator for the oxidation stability, the «induction time», is the amount of time until this marked conductivity increase occurs (Figure 2).

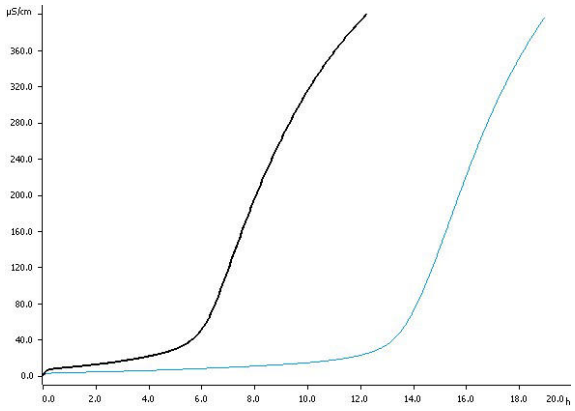

Figure 2. Determination of the oxidation stability of biodiesel both with (blue curve) and without (black curve) added antioxidants (100 mg/L ascorbyl palmitate) at 110 °C.

Table 1. Results for the oxidation stability of biodiesel with and without added antioxidants (100 mg/L ascorbyl palmitate) using the 893 Professional Biodiesel Rancimat at 110 °C.

Sample (n = 4)	Mean value in h	SD(rel) in %
Biodiesel without added antioxidant	6.15	1.1
Biodiesel with added antioxidant	13.55	0.9

CONCLUSION

The oxidation stability of biodiesel as well as blends of biodiesel is an important quality control parameter in a series of standards defining the minimum quality requirements for FAME (fatty acid methyl ester) analysis in biodiesel marketed as vehicle fuel or heating oil.

In addition, a comparison of biodiesel with and without the presence of added antioxidants can be made by determining oxidation stability according to EN 15751. This allows conclusions to be drawn about the shelf life, the effect of antioxidants, durability, and comparative value between samples which are still stable and those which are already rancid.

With the Rancimat, this quality parameter can easily and simultaneously be determined for eight different samples at a time, increasing quality control laboratory throughput. The status of the Rancimat is shown with a built-in display. Buttons for each measuring position on the instrument allow individual measurements to be started. The use of practical disposable reaction vessels and dishwasher-safe accessories reduces cleaning to a minimum. This saves time and money and significantly improves accuracy and repeatability.

Biodiesel samples with and without antioxidant addition (100 mg/L ascorbyl palmitate) have been tested in this Application Note. The determination worked superbly and has shown that biodiesel with antioxidant additives has a significantly higher induction time and therefore longer shelf life.

REFERENCES

1. Metrohm AG. Oxidation Stability of Diesel, Biodiesel, and Blends – Reliable Oxidation Stability Measurements in Diesel, Biodiesel, and Blends According to EN 14112, EN 15751, and EN 16568; AN-R-034; Metrohm AG: Herisau, Switzerland, 2024.

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CONFIGURATION

893 Professional Biodiesel Rancimat

The 893 Professional Biodiesel Rancimat is an analysis system for easy and safe determination of the oxidation stability of biodiesel (fatty acid methyl ester, FAME) and biodiesel blends in accordance with EN 14112, EN 15751 and EN 16568 standards. With eight measuring positions in two heating blocks. The built-in display shows the status of the instrument and each individual measuring position. Start buttons for every measuring position enable the measurement start on the instrument. Cleaning effort can be reduced to a minimum through the use of practical disposable reaction vessels and dishwasher-safe accessories. This saves time and costs and significantly improves accuracy and reproducibility.
All accessories necessary for carrying out determinations are included in the scope of delivery. The StabNet software is required for instrument control, data recording and evaluation and for data storage.

Equipment for determination of temperature correction with Biodiesel Rancimats.

Set for exact temperature adjustment

Consumable Kit Biodiesel Rancimat

Assembly of important expendable items for the Biodiesel Rancimat.