

Xenemetrix

The Power to Change Energy Into Information



Application note issue #7 - Quantitative analysis of Sulfur in Biodiesel June 2018

Quantitative analysis of Sulfur in Biodiesel using EDXRF portable analyser P-Metrix

EDXRF using P-Metrix instrument, is a well suited, reliable and consistent method for Sulfur measurement at very low concentrations, meeting all regulatory requirements.

BACKGROUND

EDXRF is a quick non-destructive technique for qualitative elemental analysis of main & trace elements and quantitative for all elements content in solids, powders or liquids. Having the advantage of being extremely quick, easy to operate and requiring hardly no sample preparation has increased its popularity among diverse industries such as Chemicals, Mines & Geology, Petrochemical, Polymer industry.

Biodiesel refers to vegetable oil or animal fat-based diesel fuel consisting of long-chain alkyl (methyl, ethyl, or propyl) esters. Biodiesel is typically made by chemically reacting lipids (like vegetable oil, soybean oil, animal fat with alcohol producing fatty acid esters).

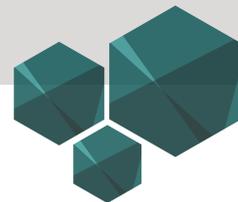
The technical definition of biodiesel is a fuel suitable for use in compression ignition. It is important to measure the amount of Sulfur because it is a pollutant and Restricted.

According to regulation Standards, American and European, the restricted Sulfur concentration is 15, 10 PPM respectively.

System configuration:

Xenemetrix system	P-Metrix
Instrument Configuration	Ag-anode X-ray tube
Detector	Silicon Drift Detector (SDD)
Atmosphere	Helium
Acquisition time	sec 600
Analysis method	Quantitative
Sample Preparation & Details	No sample preparation is required in this case





OBJECTIVE

- P-Metrix SDD calibration for quantitative elemental analysis of Sulfur in biodiesel.
- Obtaining the capability of routine quantitative analysis of Sulfur in biodiesel, Where the main goal is to measure the concentration of Sulfur at very low concentration range 2-40 PPM.

EXPERIMENTAL and RESULTS

- Six Sulfur in biodiesel samples were provided. Calibration curve was established with P-Metrix EDXRF analyzer.
- The samples, well shaken, were transferred into XRF sample cups with prolene (4 m) film support. Acquisition parameters: 600 seconds per sample.
- Helium purge was used to replace the air atmosphere, in the X-ray beam path, otherwise the air absorbs the low energy signal emitted by the Sulfur in the sample.
- Calibration curve, concentration versus intensity are plotted in a graph -Figure 2 (below). The correlation data of each concentration is in Table 2.

Figure 1 Over lapped spectra of all calibration samples

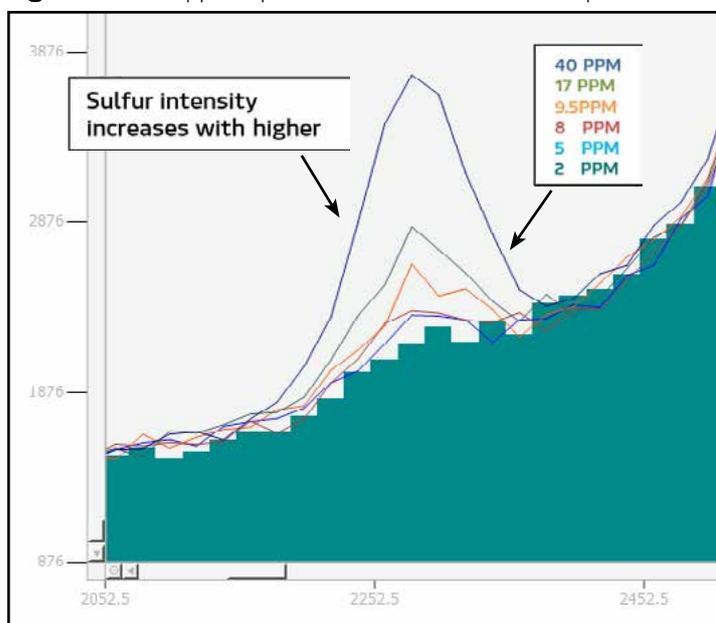


Figure 2 Regression plot, concentration versus intensity

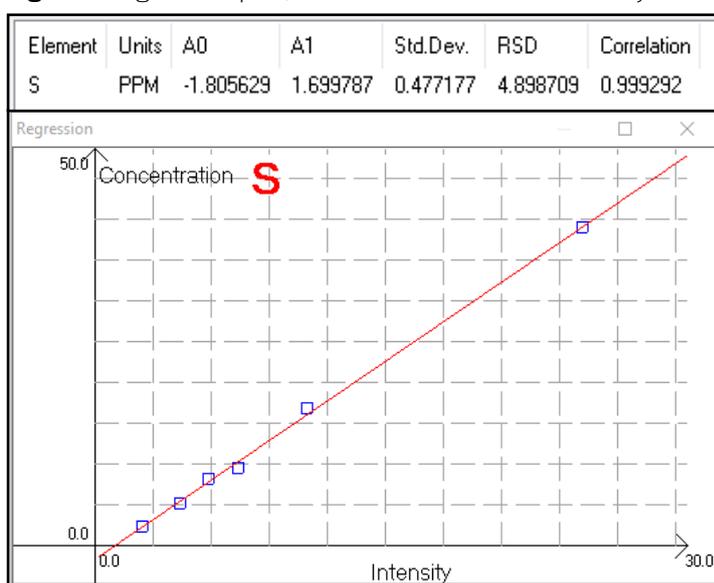
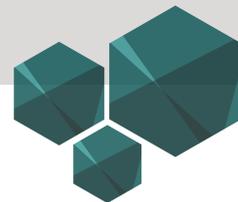


Table 2 Results of calibration regression

Given concentration (PPM)	Calculated concentration (PPM)	Absolut deviation (PPM)	Relative deviation (%)
2	1.99	0.01	0.4 %
5	5.22	0.22	4.4 %
8	7.67	0.33	4.1 %
9.5	10.25	0.75	7.9 %
17	16.21	0.79	4.6 %
40	40.16	0.16	0.4 %

Repeatability test

9.5 PPM standard sample was tested repeatedly 10 times, the result deviation from the average was ± 0.7 PPM.



DISCUSSION and CONCLUSION

P-Metrix with SDD detector is a very reliable method of measuring Sulfur in biodiesel medium, EDXRF is a proven technique of quantifying Sulfur in biodiesel samples.

Sulfur is well detected within a short period of time (Figure 1), with well resolved peaks handled by the software system.

The calibration results (Figure 2) show very good correlations between Sulfur concentration and its intensity, RSD 4.9%, STD ± 0.48 PPM, $R^2=0.99$.

In this range of concentration 5-40 PPM, EDXRF P-Metrix provides very good results.

Referring to the US and EU Biodiesel regulation, the results meet all restriction demands.

Repeatability results, ± 0.7 PPM for 9.5 PPM standard sample, demonstrated that EDXRF method meets the regulatory requirements.

Looking at the comparison between given concentration versus calculated concentration (Table 2) which is very good, obviously EDXRF using P-Metrix instrument, is a well suited, reliable and consistent method for Sulfur measurement at very low concentrations, meeting all regulatory requirements.



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