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Over the last few years, the production and use of biodiesel has increased dramatically. Pure biodiesel (B-100) contains no petroleum diesel but is blended with petroleum diesel in some cases. It can be used in conventional diesel engines without significant modifications. Biodiesel is biodegradable, non-toxic and provides significant reduction of pollutants such as sulfur emissions and aromatics. It is considered an important alternative fuel source.

ASTM has adopted a specification for pure biodiesel (ASTM D6751) that is to be used in blends up to 20% with diesel fuel. Properties such as flash point, water and sediment, carbon residue and free glycerin along with sulfur and phosphorus content are included. The current version (ASTM D6751-07b) also requires total Ca / Mg and Na / K to be determined (1). Concentration of these six inorganic components can effectively be determined using ICP-OES techniques.

A simultaneous CCD detector ICP-OES system was utilized to determine S, P, Ca, Mg, Na, and K plus 15 additional elements in biodiesel. Method parameters and results are presented (2).

Experimental conditions

The Varian 725-ES (radially viewed plasma) simultaneous CCD ICP-OES was utilized in this study. The 725-ES provides continuous wavelength coverage from 167 to 785 nm and features a high efficiency 40 MHz RF generator and low gas consumption.

The instrument operating parameters were optimized for routine organic matrix analysis. Sample introduction components included a SeaSpray nebulizer and Twister double-pass cyclonic spray chamber.

Fitted background correction automatically chooses points and requires no extra user input. The FACT peak modeling approach uses spectral data from analyte and interference standards to de-convolve the analyte peak from nearby interference peaks.

The B-100 biodiesel sample was obtained from a local biodiesel distributor (Chicago, IL). Multi-element standards

Analysis of Biodiesel Utilizing a Simultaneous CCD Detector ICP-OES System

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were prepared at concentrations of 1.0, 2.5 and 5.0 ppm from 500 ppm metallo-organic standards (S-21 + K from Conostan; Sulfur in oil from Spex CertiPrep) - diluted by weight in base oil 20. The B-100 sample was spiked at 1.0 and 2.5 ppm to measure analyte recovery.

Standards and duplicate B-100 biodiesel samples were diluted 1:10 in kerosene (Fluka purum from Sigma-Aldrich) prior to analysis.

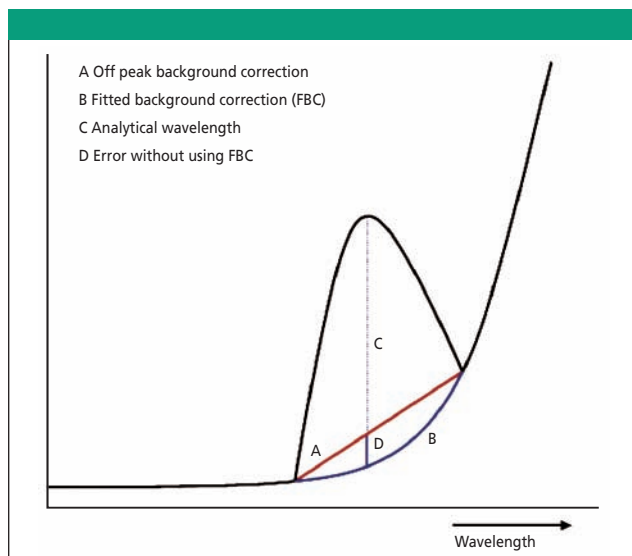


Figure 1: Fitted background correction calculates the true background signal, improving accuracy.

Table II: ASTM Specified Elements in Biodiesel

Element	P	S	Na	K	Ca	Mg
Wavelength (nm)	213.618	181.972	589.592	766.491	393.366	279.553
B-100a ppm	< IDL	1.695	0.162	0.507	0.013	0.011
B-100b ppm	< IDL	1.710	0.130	0.469	0.012	0.012
B-100+1.0 ppm (%R)	105	103	104	107	105	104
B-100+2.5 ppm (%R)	102	106	102	104	103	101
IDL (ppb)	19.9	76.5	4.1	21.8	0.1	0.1
Bkgd Correction	Fitted	Fitted	FACT	FACT	Fitted	Fitted

Table I: Instrument Parameters

Plasma Power	1.3 kW
Nebulizer Flow	0.55 L/min
Replicate Read Time	10 sec
Number of Replicates	3
Pump Rate	10 rpm
Background Correction	Fitted or FACT

