Fast, Accurate FAMEs Analyses of Biodiesel Fuel

Using a Stabilwax® Capillary GC Column

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- Stable baselines, excellent peak symmetry, baseline resolution of all compounds.
- Analysis complete in less than 11 minutes.
- All RSD% values less than 1%.

A Stabilwax® fused silica GC column affords excellent peak symmetry, resolution, and reproducibility for determining the fatty acid methyl ester (FAME) and linolenic acid methyl ester content in B100 biodiesel fuel, using European standard method EN 14103. The chromatograms and quantified data shown here were generated from four different sources of biodiesel fuel, and meet or exceed the method criteria.

As biodiesel fuel continues to stimulate interest worldwide as an energy source, several gas chromatographic methods have been developed to determine the quality of B100 fuel. European standard method EN 14103 is used for determining the FAME and linolenic acid methyl ester content, European standard method EN 14105 and ASTM standard method D-6584-00e1 are used for determining free and total glycerin, and European standard method EN 14110 is used for determining residual methanol. Method EN 14103 permits the analyst to assure the B100 product is greater than 90% fatty acid methyl esters (m/m) and the linolenic acid content is between 1% and 15% (m/m). The analysis is appropriate for FAME compositions between C14:0 and C24:1.

In evaluating the suitability of the Stabilwax® column for quantifying FAMEs and linolenic acid methyl ester by method EN 14103, we prepared reference standards from each of the four B100 fuel sources – soy, tallow, rapeseed, and yellow grease (Table 1) – by weighing 250mg of the source material into a 10mL vial, then adding 5mL of a 10mg/mL solution of internal standard methyl heptadecanoate. (Avoid allowing the samples to stand longer than 12 hours, or quantification will be inaccurate.) We installed the 30m x 0.32mm ID x 0.25µm Stabilwax® column (cat.# 10624) in an Agilent 6890 instrument equipped with a split/splitless injector, a flame ionization detector, and ChemStation software. To obtain the fastest analysis, without sacrificing resolution, we selected hydrogen as the carrier gas, supplied from a Parker Balston hydrogen generator.

Figure 1 shows, for each source material, the analysis to FAME C24:1 is completed in less than 11 minutes. Particularly notable are the stability of the baselines, the excellent peak symmetry, and baseline resolution of all compounds of interest. Table 2 summarizes the RSD% values for the FAMEs measurements, all of which are less than 1%.

A 30m x 0.32mm ID x 0.25µm Stabilwax® column, used with hydrogen carrier gas, permits high speed analysis and ensures precise data acquisition for accurate quantification of C14:0-C24:1 FAMEs and linolenic acid methyl ester.

Table 1 Sources of FAMEs in B100 biodiesel fuel (% m/m).

		Soy	Tallow	Rapeseed	Yellow Grease
Myristic acid	C14:0	0.21	1.7	0.11	0.68
Palmitic acid	C16:0	11.24	25.5	4.1	16.35
Palmitoleic acid	C16:1	0.2	3.27	0.27	1.23
Stearic acid	C18:0	4.04	14.41	1.8	9.32
Oleic acid	C18:1	21.93	40.34	58.57	47.8
Linoleic acid	C18:2	53.84	12.02	22.2	20.01
Linolenic acid	C18:3	7.29	0.99	13.26	2.93
Arachidic acid	C20:0	0.36	0.4	0.79	0.46
Gadoleic acid	C20:1	0.26	1.03	1.79	0.39
Behenic acid	C22:0	0.45		0.57	0.44
Erucic acid	C22:1			0.13	0.23
Lignoceric acid	C24:0	0.16	0.34	0.3	0.24
Nervonic acid	C24:1		0.17	0.54	

Table 2 Relative standard deviations for FAMEs do not exceed 1% in analyses on a Stabilwax® column (n = 3).

		Soy	Tallow	Rapeseed	Yellow Grease
Myristic acid	C14:0	0.33	0.42	0.24	0.36
Palmitic acid	C16:0	0.04	0.06	0.02	0.04
Palmitoleic acid	C16:1	0.23	0.17	0.19	0.09
Stearic acid	C18:0	0.05	0.02	0.13	0.19
Oleic acid	C18:1	0.02	0.3	0.2	0.25
Linoleic acid	C18:2	0.25	0.41	0.11	0.22
Linolenic acid	C18:3	0.13	0.16	0.07	0.14
Arachidic acid	C20:0	0.3	0.37	0.23	0.31
Gadoleic acid	C20:1	0.33	0.28	0.37	0.41
Behenic acid	C22:0	0.28		0.29	0.17
Erucic acid	C22:1			0.21	0.26
Lignoceric acid	C24:0	0.53	0.14	0.1	0.33
Nervonic acid	C24:1		0.55	0.83	

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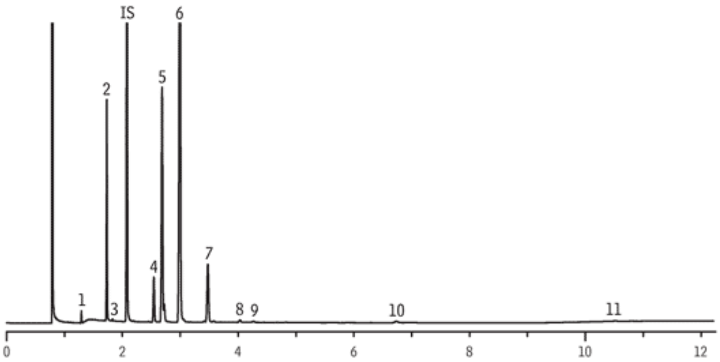
Figure 1 Stable baselines, excellent peak symmetry, and rapid, baseline resolution of all compounds characterize FAMEs analyses on a Stabilwax® column.

Soy

1.	myristic acid	C14:0	6.	linoleic acid	C18:2
2.	palmitic acid	C16:0	7.	linolenic acid	C18:3
3.	palmitoleic acid	C16:1	8.	arachidic acid	C20:0
	methyl heptadecanoate (IS)		9.	gadoleic acid	C20:1
4.	stearic acid	C18:0	10.	behenic acid	C22:0
5.	oleic acid	C18:1	11.	lignoceric acid	C24:0

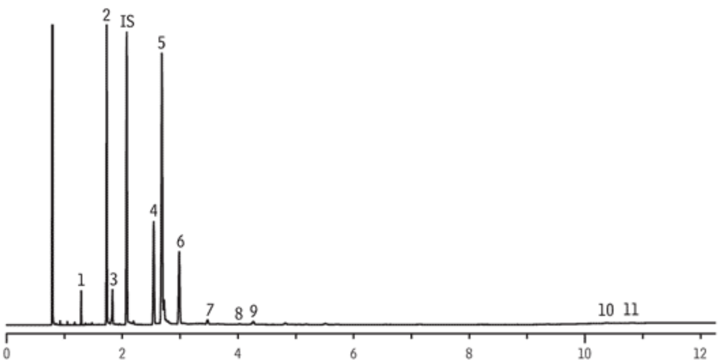


Sample: soy source of biodiesel (B100), prepared according to European Method EN 14103

GC_PC00915

Tallow

1.	myristic acid	C14:0	6.	linoleic acid	C18:2
2.	palmitic acid	C16:0	7.	linolenic acid	C18:3
3.	palmitoleic acid	C16:1	8.	arachidic acid	C20:0
	methyl heptadecanoate (IS)		9.	gadoleic acid	C20:1
4.	stearic acid	C18:0	10.	lignoceric acid	C24:0
5.	oleic acid	C18:1	11.	nervonic acid	C24:1

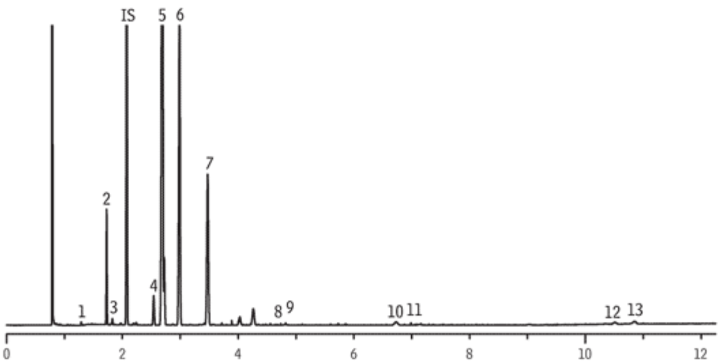


Sample: tallow source of biodiesel (B100), prepared according to European Method EN 14103

GC_PC00916

Rapeseed

1.	myristic acid	C14:0	7.	linolenic acid	C18:3
2.	palmitic acid	C16:0	8.	arachidic acid	C20:0
3.	palmitoleic acid	C16:1	9.	gadoleic acid	C20:1
	methyl heptadecanoate (IS)		10.	behenic acid	C22:0
4.	stearic acid	C18:0	11.	erucic acid	C22:1
5.	oleic acid	C18:1	12.	lignoceric acid	C24:0
6.	linoleic acid	C18:2	13.	nervonic acid	C24:1

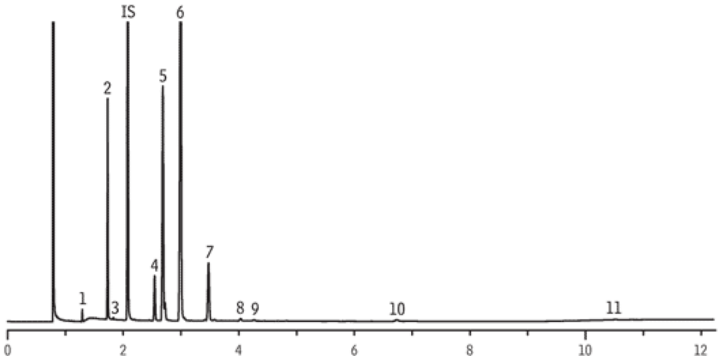


Sample: rapeseed source of biodiesel (B100), prepared according to European Method EN 14103

GC_PC00917

Yellow Grease

1.	myristic acid	C14:0	7.	linolenic acid	C18:3
2.	palmitic acid	C16:0	8.	arachidic acid	C20:0
3.	palmitoleic acid	C16:1	9.	gadoleic acid	C20:1
	methyl heptadecanoate (IS)		10.	behenic acid	C22:0
4.	stearic acid	C18:0	11.	erucic acid	C22:1
5.	oleic acid	C18:1	12.	lignoceric acid	C24:0
6.	linoleic acid	C18:2			



Sample: yellow grease source of biodiesel (B100), prepared according to European Method EN 14103

GC_PC00918

Column:	Stabilwax®, 30m, 0.32mm ID, 0.25µm (cat.# 10624)
Inj.:	1.0µL split (split ratio 100:1), Cyclosplitter® inlet liner (cat.# 20706)
Inj. temp.:	250°C
Carrier gas:	hydrogen, constant flow, 3mL/min.
Linear velocity:	60cm/sec.
Oven temp.:	210°C (hold 5 min.) to 230°C @ 20°C/min. (hold 5 min.)
Det.:	FID
Det. temp.:	250°C