Ester content Analysis in Biodiesel

1. Analytical Condition

YL 6500 Series

Oven : 200℃(for 8min)->10 ℃ /min ->230 ℃(for 10min)

Capillary Column : HP-Innowax(30m*0.32mm*0.25um)

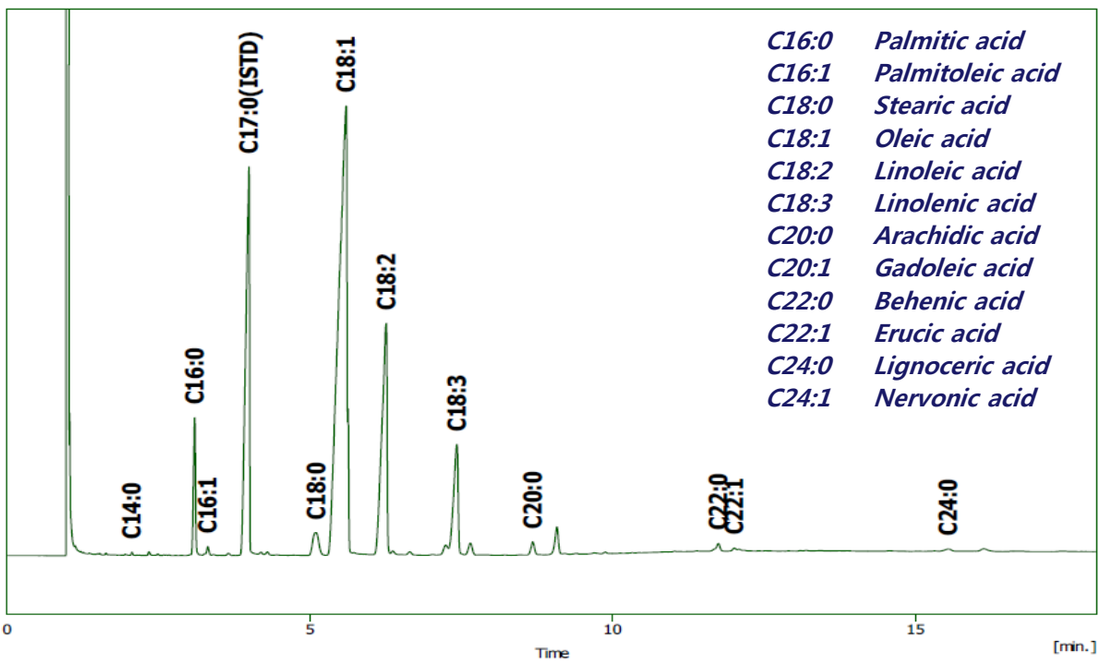
Injector : Capillary 250℃

Column flow : 1-3ml/min

Detector : FID 250 ℃

Split ratio 10:1-50:1

2. Chromatogram



Fatty Acid Analyzer

In the food industry, the quality management for products is conducted by analyzing esterified fatty acids to determine the ratio of saturated and unsaturated fatty acids in fat.

There are numerous kinds of fatty acid analysis such as fatty acids in edible oils (ex-sesame oils), recently brought up social issue- trans fatty acids, omega 3 fatty acids (refined fish oils) in healthy functional food, etc.

For fatty acids have many isomers to be separated, they are commonly analyzed by gas chromatograph after esterified to Fatty Acid Methyl Esters (FAMEs) to avoid peak tailings by a direct injection of fatty acid and column clogging.

It's very important to set an optimal oven program because the polar or moderately polar capillary columns used in this analysis have low temperature limit.

The recommended column for the analysis of fatty acids in edible oils is polar capillary columns such as Omegawax of Innowax, and for trans fatty acids is moderately polar capillary columns such as HP-23 or SP-2560 to separate cis/trans isomers. The order of peaks must be indentified first for the eluted peaks of fatty acids are different in the use of polar or moderately polar capillary columns.

YL Fatty Acid Analyzer is suitable to analyze various fatty acids and provide all the solution from preparation of sample to analysis.

• Useful Information

1) Sample preparation

(1) Sample

- **Lipolysis :**
  Sample 25mg ➞ Add 0.5N **NaOH methanol** solution 1.5 ml and mix
  ➞ Heat at 100 °C for 5 min ➞ Cool down to 30-40 °C

- Fatty Acids Esterification:
  ➞ Add 14 % Trifluoroborane methanol solution 2 ml and mix
  ➞ Heat at 100 °C for 2 min
  ➞ Cool down to 30-40 °C, add 1 ml of hexane (or heptane) and stir it for 30 sec.
  ➞ Add 5 ml of saturated NaCl solution and stir
  ➞ Separate layers at the ambient temperature
  ➞ Use an upper layer (Hexane or hexane layer) as a sample

(2) Standard

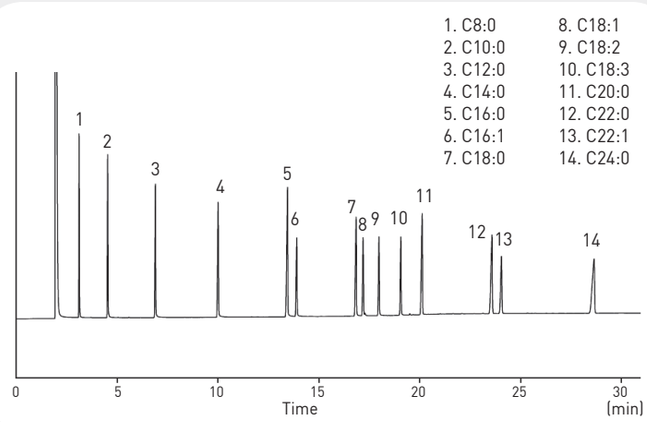
Fatty acids in edible oils –14 kinds of Fatty acid methyl ester mix
Trans fatty acids- 37 kinds of Fatty acid methyl ester mix
cis, trans isomer standard of 18:2, 18:3
Omega 3 fatty acids – DHA, EPA standard

• Application

- Fatty acids in edible oils
- Trans fatty acids
- Omega 3 fatty acids (DHA, EPA)

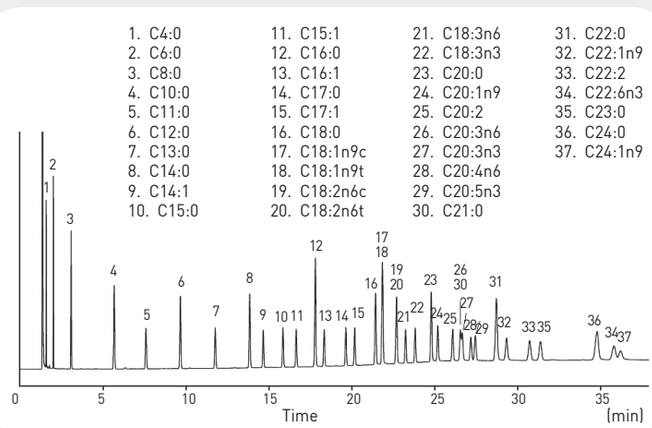
■ Fatty Acid Methyl Esters (FAMEs)

- Oven : 140 °C (1 min) ➞ 5 °C/min ➞ 240 °C (5 min)
- Column : HP-INNOWAX (30 m, 0.25 mm, 0.25 μm)
- Carrier gas : He, 1.0 ml/min (Split ratio 20:1)
- Injector : Capillary 250 °C
- Detector : FID 250 °C
- Injection volume : 1 μl (Liquid)



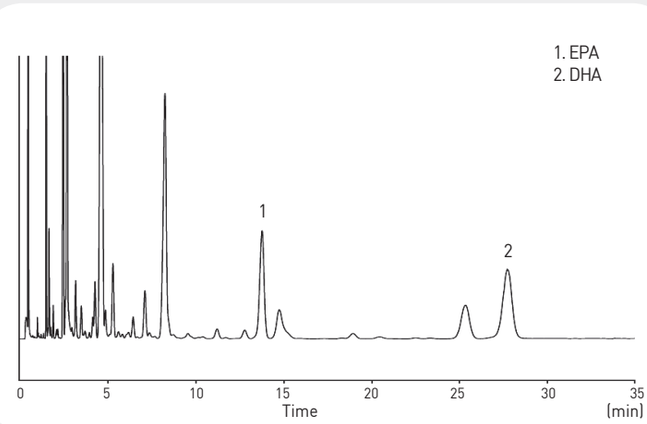
■ Fatty Acid Methyl Esters (FAMEs)

- Oven : 140 °C (5 min) ➞ 5 °C/min ➞ 240 °C (20 min)
- Column : HP-INNOWAX (30 m, 0.53 mm, 1.0 μm)
- Carrier gas : He, 5.0 ml/min (Split ratio 20:1)
- Injector : Capillary 250 °C
- Detector : FID 250 °C
- Sample : Fatty Acid Methyl Esters
- Injection volume : 1 μl (Liquid)



■ DHA & EPA in Refined Fish Oils

- Oven : 200 °C (Isothermal)
- Column : HP-INNOWAX (30 m, 0.53 mm, 1.0 μm)
- Carrier gas : He, 7.0 ml/min (Split ratio 10:1)
- Injector : Capillary 250 °C
- Detector : FID 250 °C
- Sample : Refined Fish (Preparation : FAME)
- Injection volume : 1 μl (Liquid)



VOCs Analyzer

YL VOCs Analyzer accurately tests for the presence of volatile organic compounds (VOCs) which may be dissolved in drinking and waste water supplies. The Analyzer operates by first purging VOCs from a water sample under pressure using inert gasses such as helium or nitrogen. The VOCs are trapped on a solid carbon-based sorbant in a trap tube and are concentrated by thermal de-sorption, and then separated by passing the VOCs through a column in a column oven. The separated volatile constituents are qualitative and/or quantitative detected with either the flame ionization detector (FID) or electron capture detector (ECD).

Of the about 300 VOCs known, 70 are easily dissolved in water; of these 70 VOCs, 18 are regulated by the Korean Ministry of the Environment for meeting drinking water quality standards (KDWR and the EPA 500 series method).

YL VOCs Analyzer accurately tests for the presence of VOCs and measures their concentration. The system contains all necessary reagents and equipment for conducting the analyses, including detailed procedures and protocols for conducting the tests. These protocols are in full compliance with approved U.S. EPA methods.

• Useful Information

1) Sample Collection

Compounds	Container	Additives	Storage
Volatile Organic Compounds	Glass/Brown bottle	Ascorbic acid or Sodium Thiosulfate	4 °C

VOCs are collected as a sample in a 40 ml vial. Collection must be done carefully so as to avoid any empty space or air bubble in the sample vial, which would interfere with the accuracy of downstream measurements of VOCs in the sample. The collected sample may be stored at 4 °C for up to 14 days, but it is recommended analyses of the collected sample be conducted as soon as possible but no later than 14 days of sampling.

2) Sample preparation after collecting the sample

Add ascorbic acid to the collected sample in the 40 ml vial. Ascorbic acid prevents the oxidation of any VOCs present in the sample. Such oxidation would be THM in the presence of Humic acid, Fluvic acid and Choloride. Adjust the pH of the sample to pH 2.0 or less with 6N HCl. Store the sample in a dark location until ready to proceed with the analysis.
If using the Purge & Trap method, inject the thermal-desorbed compound to the Oven system module. Proceed with the detection and measurement of VOCs that may be present in the sample.

3) Preparation of Calibration Standards

Use the standards included in the kit. In order to make a VOC blank solution and standards, it is suggested that ultra-pure water (YL aquaMAX Ultra System) be used. Prepare the water by first the heated water passing with 99.995 % of Nigrogen gas for one hour, and then the water is allowed to cool passing with Nitrogen gas, which is a VOC blank solution. Using the prepared blank solution, dilute the highly concentrated standards based on the calibration of concentration standards to make a standard.

4) Detectors Used in the Analysis of VOCs

We recommend the use of the detectors listed in [Table 1] when testing for the presence of the 18 volatile organic compounds, which are restricted by KDWR regulations for water quality standards set for drinking water.

	Targeted compounds	Detectors suggested by regulation of water quality for drinking water
1	Phenol	UV
2	THM	FID, ECD, ELCD, PID
3	1.1.1-Trichloroethane	FID, ECD, ELCD, PID
4	Tetrachloroethylene	ECD
5	Trichloroethylene	ECD
6	Dichloromethane	ECD
7	Benzene	FID, ECD, ELCD, PID
8	Toluene	FID, ECD, ELCD, PID
9	Ethylbenzene	FID, ECD, ELCD, PID
10	Xylenes (-o, -p, -m)	FID, ECD, ELCD, PID
11	1.1-Dichloroethylene	FID, ECD, ELCD, PID
12	Carbon tetrachloride	FID, ECD, ELCD, PID
13	1.2-dibromo-3-chloropropane	ECD, Mass
14	Chloralhydrate	ECD, Mass
15	Dibromoacetonitrile	ECD, Mass
16	Dichloroacetonitrile	ECD, Mass
17	Trichloroaceetonitrile	ECD, Mass
18	Haloaceticacid (6 kinds)	ECD, Mass

[Table 1] Detectors are recommended for testing the presence of VOCs restricted by the KDWR water quality regulations established for drinking water supplies

• Application

- Sterilized by-product in water
- VOC in drinking water

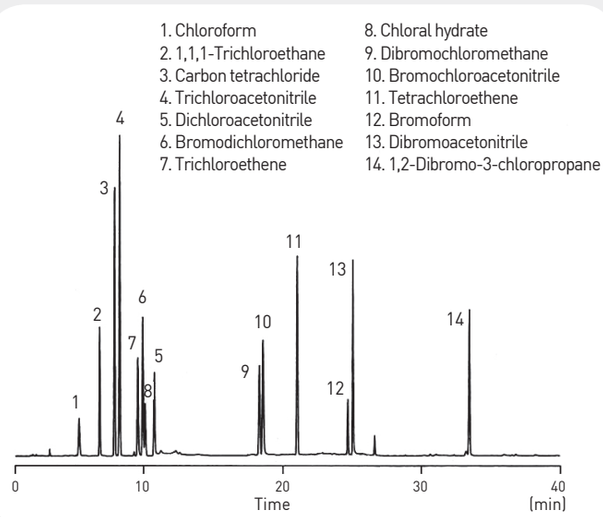
■ Halogenated Compounds

- Injection transfer line : 280 °C
- Column : HP-VOC (60 mm, 0.32 mm, 1.8 μm)
- Oven temperature program

5 °C/min 7 °C/min

35 °C (5 min) 60 °C (0 min) 200 °C (10 min)

- Purge & Trap :
  - -Trap : Charcoal/Tenax/Silica
  - -Purge flow : 40 ml/min
  - -Purge time : 7 min
  - -Desorption temp : 225 °C
  - -Desorption time : 4 min
  - -Injection temp : 180 °C
  - -Injection time : 1 min
- Detector : ECD, 250 °C
- Carrier gas : N2



Residual Solvent Analyzer

Organic residual solvents used in the manufacture of pharmaceuticals and found in the inks used for the printing of packaging materials for food and drug products are known to be hazardous to human health if ingested. YL Residual Solvent Analyzer can accurately and efficiently detect and quantify residual solvents.

• Useful Information

• Sample Injection Condition

*** Pharmaceutical raw materials**

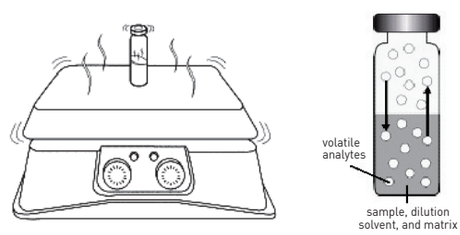
Incubate the vial containing the standard and the sample in the Headspace Autosampler in a dry oven for 60 minutes at 85 °C. Sample 1.0 - 1.5 ml of above the gas phase from the vial with a gastight syringe and inject it.
Note that the incubation temperature and time are flexible, which is based on the boiling points of the organic solvents.

*** Food packaging materials**

Inject 1.0 – 1.5 ml of the gas phase sampled by a gastight syringe after heating the tripod flask containing the standards and the sample in a dry oven for 30 minutes at 80 °C.

• Automation

Headspace Autosampler can make automation of sample preparation and Injection to the Residual Solvent Analyzer.



[Fig.1] Heating of the standard and sample

• Recommended Columns

- Food packaging material: G1 or G5 (30 m, 0.32 mm, 1.0um)
- Pharmaceutical raw materials: G16 (30 m, 0.53 mm, 1.0 μm), G43, G1

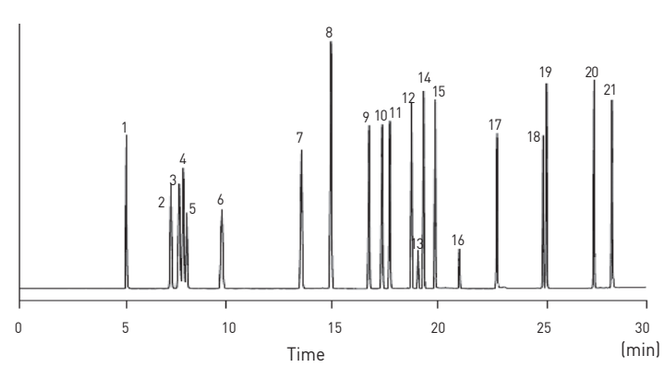
• Application

- Residual solvents in food packaging materials
- Residual solvents in pharmaceutical raw materials

■ Solvent Mixture

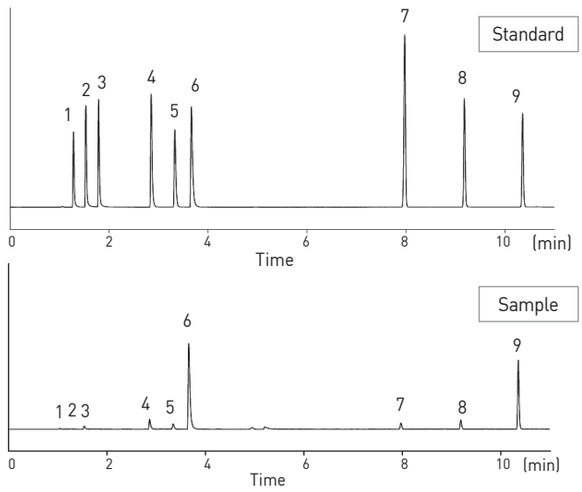
- Oven : 35 °C (10 min) ➞ 5 °C/min ➞ 160 °C (5 min)
- Column : HP-INNOWAX (60 m, 0.32 mm, 0.5 μm)
- Carrier gas : He / 1 ml (Split ratio 100:1)
- Injector : Capillary 250 °C
- Detector : FID 300 °C

1. Acetone
2. Ethyl acetate
3. Iso propyl acetate
4. Methanol
5. Methyl ethyl ketone
6. Iso-propanol
7. Methyl isobutyl ketone
8. Toluene
9. Butyl acetate
10. Isobutyl isobutyrate
11. Iso-butanol
12. p-xylene
13. Xylene impurity
14. Propylene glycol – monomethyl ether
15. Butanol
16. o-xylene
17. Ethyl cellosolve
18. Cellosolve acatate
19. Cyclohexanone
20. Diacetone alcohol
21. Butyl cellosolve



Residual Solvents in the Food Packing Materials

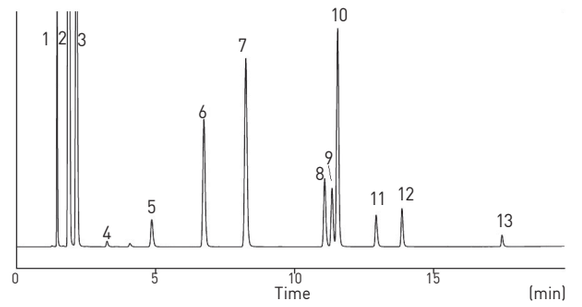
- Oven : 35 °C (5 min) ➞ 10 °C/min ➞ 150 °C (5 min)
- Column : HP-5 (30 m, 0.32 mm, 1.0 μm)
- Carrier gas : He, 3 ml/min (Split ratio 10:1)
- Injector : Capillary 230 °C
- Detector : FID 250 °C
- Headspace : 80 °C, 30 min Heating, Gas 1 ml Injection



1. MeOH
2. EtOH
3. IPA
4. MEK
5. EA
6. THF (ISTD)
7. Tol
8. BA
9. PMA

Residual Solvents on Class 2

- Oven : 35 °C (5 min) ➞ 5 °C/min ➞ 100 °C (0.1 min)
  ➞ 13 °C/min ➞ 200 °C (5 min)
- Column : HP-INNOWAX (30 m, 0.53 mm, 1.0 μm)
- Carrier gas : He, 6 ml/min (Split ratio 5:1)
- Injector : Capillary 230 °C
- Detector : FID 250 °C
- Headspace : 80 °C, 60 min Heating, Gas 1 ml Injection



1. n-Hexane
2. Cyclohexane
3. Methyl cyclohexane
4. Trichloroethylene
5. Dichloromethane
6. Dichloroethylene
7. Toluene
8. Ethyl benzene
9. p-Xylene
10. m-Xylene
11. o-Xylene
12. chlorobenzene
13. DMF

Ethanol in Hand Washes

- Oven : 50 °C (3 min) ➞ 15 °C/min ➞ 150 °C (1 min)
- Column : HP-INNOWAX (30 m, 0.53 mm, 1.0 μm)
- Carrier gas : He, 6 ml/min (Split ratio 15:1)
- Injector : Capillary 230 °C
- Detector : FID 250 °C
- Injection volume : 1 μl Direct Injection