

Analysis of Aroma Components in Essential Oils with AOC™-30i

Toshinari Ishii

User Benefits

- ◆ Regardless of the viscosity of samples, the Sampler Navigator makes it easy to set injector parameters.
- ◆ The increased number of wash vials in AOC-30i prevent carryover, solvent depletion, and sample contamination.
- ◆ The combination of multiple solvents maximizes the efficiency of syringe wash.

Introduction

Essential oils are obtained from plants and fruits through steam distillation. They are widely used as additives in cosmetics and foods. As most essential oils are highly viscous, they must be diluted for gas chromatography (GC) analysis. However, sample dilution complicates the detection of trace aroma components.

In this study, we analyzed peppermint oil without dilution by using the Sampler Navigator of AOC-30i and measured the aroma components.



Figure 1. Nexis™ GC-2030 + AOC™-30i

Solvents for syringe wash

Essential oils remain attached to the syringe needle owing to high viscosity. In addition, they are complex samples containing various components. As such, when they must be measured continuously, the syringe must be washed to reduce carryover and sample left.

AOC-30i can hold four wash vials and handle complex matrices by washing the syringe with multiple types of solvents.

For essential oils, nonpolar solvents, such as hexane, are recommended to be combined with polar solvents such as ethanol and acetone.

The increased number of wash vials also reduces solvent depletion and sample contamination.

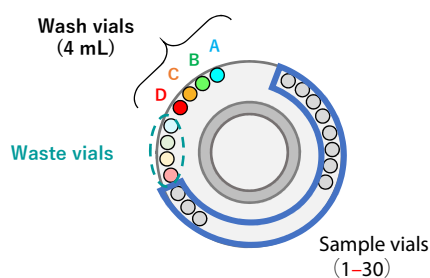


Figure 2. AOC-30i turret

Injector setting based on Sampler Navigator

A high-viscosity sample may create air bubbles in the barrels and interfere with accurate volume measurement.

Figure 3 (top) represents the lemon-oil chromatogram with the "Default" setting of AOC-30i. This figure shows that the sample was not injected fully in the 8th analysis. Here, injector parameters, such as injection speed and number of washes, must be determined.

LabSolutions™ GC preset the recommended parameters of AOC-30i for the sample type and aim of the analysis (Figure 4). Each presetting was suggested by an experienced analyst, and the recommended parameters can be set by one click.

"Inject Viscous Sample" setting is the optimum condition to inject high-viscosity samples in a stable manner. Figure 3 (bottom) shows the settings used to measure stable lemon oil.

※ This is an example of results. We cannot guarantee reproducibility, because viscosity changes with temperature and humidity

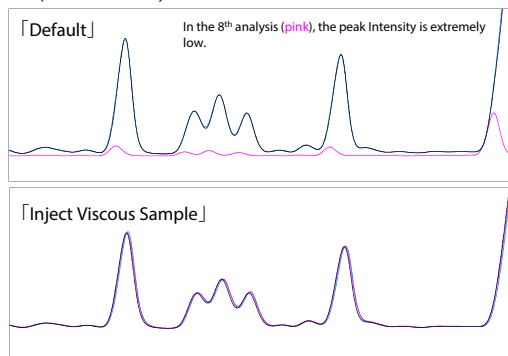
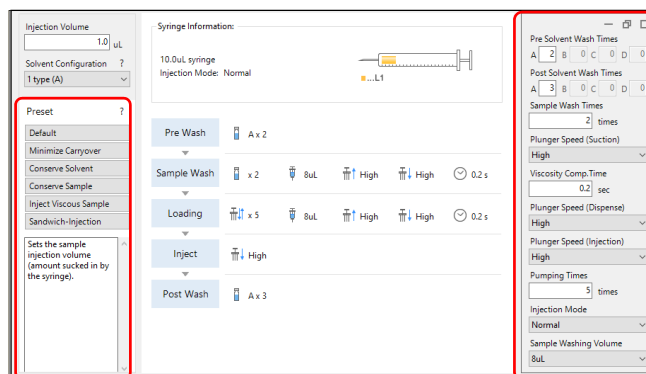


Figure 3. Lemon-oil chromatogram
(7th, 8th, and 9th results of 10 consecutive analyses)



Sampler Navigator
Select the settings for sample type and aims

Can edit the detail of each parameter
※ The original preset remain the same

Figure 4. LabSolutions™ GC

■ Analysis of Peppermint Oil

We analyzed peppermint oil with Nexis GC-2030 + AOC-30i and Xtra Life Microsyringe, 10 µL (P/N 227-35400-01).

Acetone (A) was used as the prewash solvent, with Ethanol (B), Acetone (C), and Hexane (D) as postwash solvents.

※ A → Injection → B → C → D

Table 1 Analysis condition

Model	: Nexis GC-2030 / AOC-30i	
Injection Volume	: 0.5 µL	
Injection Temp.	: 260 °C	
Injection Mode	: Split mode	
Split Ratio	: 1:100	
Carrier Gas	: He	
Carrier Gas Control	: linear velocity (40 cm/s)	
Column	: SH-Rtx™-Wax (P/N 221-75897-30) (30 m × 0.32 mm I.D., 1.0 µm)	
Column Temp	: 90 °C - 8 °C/min - 240 °C (15 min)	
Detector	: Flame Ionization Detector (FID)	
Detector Temp	: 300 °C	
Detector Gas	: H ₂ 32.0 mL/min、 Air 200 mL/min	
Makeup Gas	: He (24 mL/min)	
Injector Setting	: Pumping Times	0 times
	: Presolvent Wash Time	0 times
	: Plunger Speed (Injection)	middle
	: Plunger Speed (Suction)	middle
	: Viscosity Comp. Time	3.0 s
	※automatically set by "Inject Viscous Sample"	

■ Results

Seventeen components were identified, as shown in Figure 5. The area reproducibility of these components was 0.8% or less, showing good results (Table 2). In the "Inject Viscous Sample" setting, the operating speed of the syringe is slower than usual, and the number of times a solvent is washed is increased; thus, the analysis can be continued even in the case of highly viscous samples.

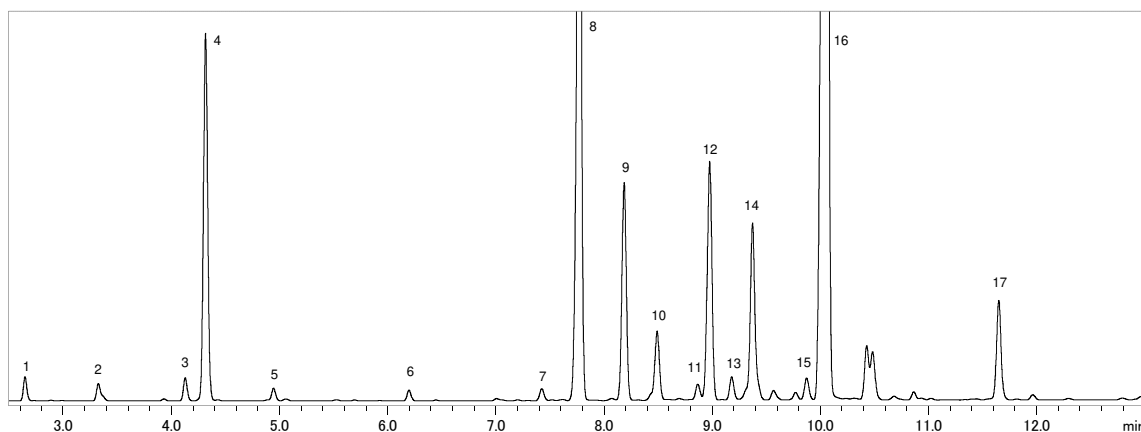


Figure 5. Chromatogram of Peppermint Oil

Nexis, AOC and LabSolutions are trademarks of Shimadzu Corporation in Japan and/or other countries.
Rtx is either a trademark or a registered trademark of Restek Corporation in the United States and/or other countries.

Table 2 Reproducibility of Aroma Components (n=5)

No.	Components	R.T. (min)	Area Reproducibility (RSD%)
1	β-Pinene	2.632	0.67
2	Sabinene	3.306	0.70
3	D-Limonene	4.107	0.68
4	Eucalyptol	4.294	0.62
5	p-Cymene	4.919	0.65
6	3-Octanol	6.173	0.63
7	trans-Sabinene hydrate	7.398	0.67
8	D-Menthone	7.748	0.61
9	D-isomenthone	8.161	0.60
10	β-Bourbonene	8.471	0.61
11	Menthyl formate	8.844	0.72
12	Menthyl acetate	8.958	0.59
13	Isopulegol	9.156	0.65
14	Neomenthol	9.352	0.63
15	Terpinen-4-ol	9.542	0.56
16	Menthol	10.038	0.61
17	Peritone	11.625	0.62

■ Summary

For analyzing essential oils without dilution, we recommend using the "Inject Viscous Sample" setting of the Sampler Navigator. A continuous analysis is possible with good reproducibility and stability.

For better reproducibility, the Plunger Speed for "Injection" and "Suction" should be increased from middle to high. At this time, ensure that the solvent is discharged smoothly from the syringe. In contrast, if a continuous analysis cannot be conducted even in "Inject Viscous Sample" setting, decrease the Plunger Speed of "Injection" and "Suction" from middle to low.

■ Cautionary Points of Viscous Sample

Some viscous samples include refractory components. Dilute such samples, when you analyze them. Be sure to dilute vegetable oils including high concentration of Triglyceride.