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Application News

GC Nexis™ GC-2030

Streamlining Sample Analysis Using Automated Injection of Internal Standards

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User Benefits

- Labor intensive sample preparation work can be reduced, thus resulting in higher lab efficiency.
- Optimized method for sandwich-injection comes pre-configured on the autoinjector, allowing anyone to preform the analysis with confidence.
- Automatic injection using the autoinjector enables error-free addition of internal standards.

■ Introduction

The internal standard method is a quantitative analysis method in gas chromatography (GC), and is widely used because it can correct for instrument sensitivity and injection volume errors. On the other hand, accurate addition of the internal standard (IS) to all samples is necessary, and more sample preparation work is required in comparison with the absolute calibration curve method (external standard method).

In this article, commonly-used alcohols, ketones, esters, ethers, and aromatics were analyzed using sandwich-injection mode of the Sampler Navigator function of the AOC™-30 autoinjector, and satisfactory linearity of the calibration curves and repeatability of the peak area ratios were verified.

■ Sampler Navigator Function

In sandwich-injection with the AOC-30 autoinjector, the syringe draws up the IS first, followed by the sample, and then simultaneously injects the two compounds into the injection port (Fig. 1). When the sandwich-injection of Sampler Navigator function is selected, the recommended conditions for automated injection of the IS can be set with one click.

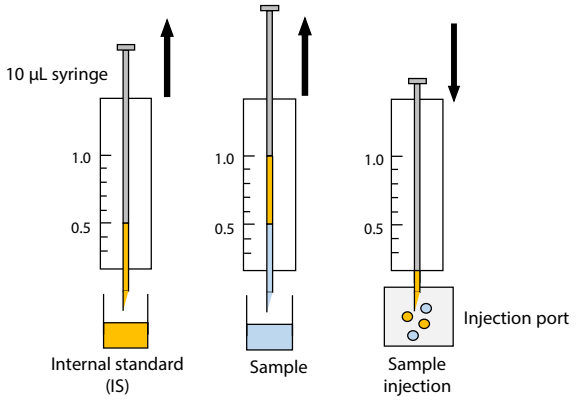

Fig. 1 Flow of Suction and Injection of IS and Sample

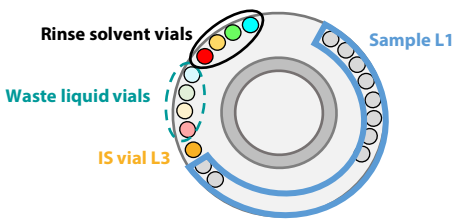

Fig. 2 Turret of AOC-30

■ Preparation of Mixed Samples

Table 1 shows the composition of the mixed samples and the IS used in this experiment. Concentrations of 20 ppm, 200 ppm, 1000 ppm, and 2000 ppm for the mixed samples were prepared by dilution with the solvents indicated in the table 1.

1000 ppm solutions of the internal standards for mixtures 1 and 2 were prepared via dilution using the respective solvents. A 1000 ppm was used for mixture 3.

Table 1 Compositions of Mixed Samples and IS and Solvents Used

	Mixture 1 (Alcohol)	Mixture 2 (Ketone, ester, ether)	Mixture 3 (Aroma)
Sample	Methanol	THF	Benzene
	2-Propanol	Ethyl acetate	Toluene
	Ethanol	MiBK	o-Xylene
	n-Propanol	MBK	Ethyl benzene
	i-Butanol	-	Tetralin
IS	2-Butanol	MEK	p-BFB
Solvent	Acetone	Acetone	Methanol

p-BFB: p-Bromofluorobenzene-Fluorobenzene Mixture Standard Solution (each 1 mg/ml Methanol Solution)

■ Conditions of Sandwich-Injection Analysis

The analysis of the mixed samples was conducted under the conditions in Table 2 using a Nexis GC-2030 + AOC-30i. The mixed samples were set at L1 in Fig. 2, and the internal standard at L3.

Table 2 Analysis Conditions

Model	: Nexis GC-2030 / AOC-30i
Injection Mode	: Sampler Navigator – sandwich-injection (IS 5 %, sample 0.5 µL) *1
Syringe	: 10 µL syringe for AOC (P/N 221-34618)
Injection Volume	: 0.5 µL (IS) + 0.5 µL (mixture)
Injection Temp.	: 200 ˚C
Injection Mode	: Split
Split Ratio	: 1: 30 (Mixture 1, 3) 1: 50 (Mixture 2)
Carrier Gas	: He
Carrier Gas Control	: Linear velocity (35 cm/s)
Column	: SH-Stabilwax™ (P/N 227-36252-02) (60 m x 0.32 mm I.D., 1.0 µm)
Column Temp.	
Mixture1	: 70 ˚C (5 min) - 5 ˚C/min - 80 ˚C - 15 ˚C/min - 140 ˚C (5 min)
Mixture2	: 60 ˚C (5 min) - 10 ˚C/min - 80 ˚C - 40 ˚C/min - 140 ˚C (5 min)
Mixture3	: 90 ˚C - 20 ˚C/min - 200 ˚C(5 min)
Detector	: Hydrogen flame ionization detector (FID)
Detector Temp.	: 250 ˚C
Detector Gas	: H_2 32 mL/min, Air 200 mL/min
Makeup Gas	: N_2 24 mL/min

*1 The IS injection volume can be selected for 5 % or 10 % of the syringe volume.

■ Results

Figs. 3, 4, and 5 show the chromatograms of mixtures. Tables 3, 4, and 5 show the area ratio repeatability and R^2 values of the calibration curves for the concentration of 200 ppm.

The results show that $R^2 > 0.9995$ could be obtained for all compounds measured in this experiment, indicating that excellent linearity could be obtained. Excellent results were also obtained for repeatability of the area ratio.

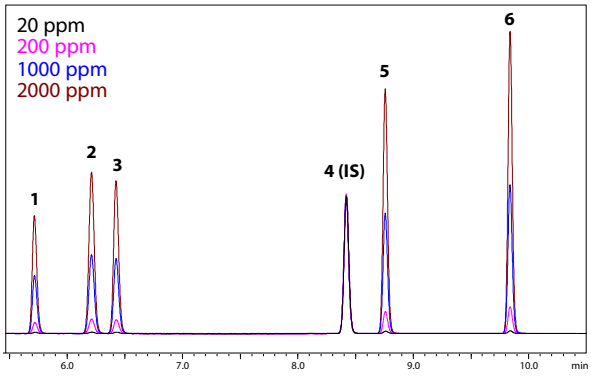

Fig. 3 Chromatogram of Mixture 1

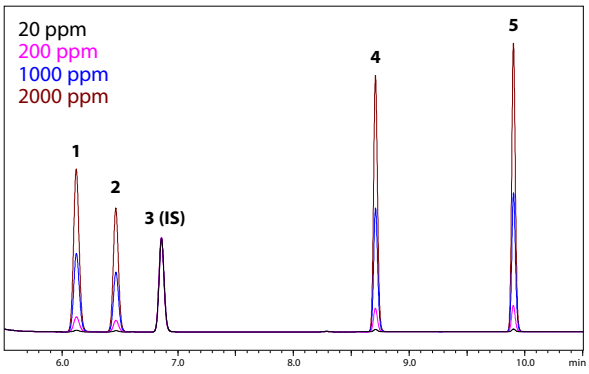

Fig. 4 Chromatogram of Mixture 2

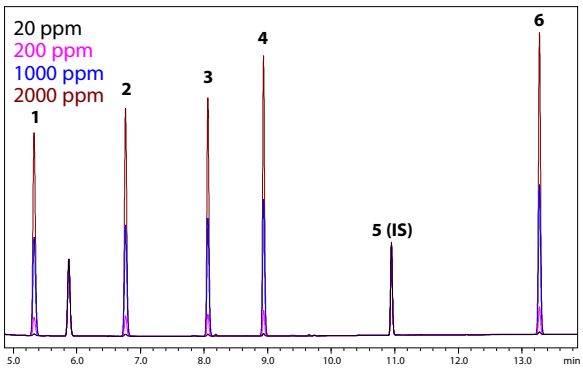

Fig. 5 Chromatogram of Mixture 3

Table 3 Retention Time, Area Ratio Repeatability, and R^2 Value of Compounds in Mixture 1 (200 ppm, n=6)

No.	Sample	Retention time (min)	Area ratio repeatability (RSD%)	R^2
1	Methanol	2.724	1.07	0.9998
2	2-Propanol	6.213	0.77	0.9998
3	Ethanol	6.428	0.74	0.9998
4	2-Butanol	8.420	-	-
5	n-Propanol	8.759	0.93	0.9998
6	i-Butanol	9.839	0.99	0.9998

Table 4 Retention Time, Area Ratio Repeatability, and R^2 Value of Compounds in Mixture 2 (200 ppm, n=6)

No.	Sample	Retention time (min)	Area ratio repeatability (RSD%)	R^2
1	THF	6.125	0.49	0.9997
2	Ethyl acetate	6.467	0.32	0.9998
3	MEK	6.857	-	-
4	MiBK	8.710	0.53	0.9997
5	MBK	9.902	0.33	0.9997

Table 5 Retention Time, Area Ratio Repeatability, and R^2 Value of Compounds in Mixture 3 (200 ppm, n=6)

No.	Sample	Retention time (min)	Area ratio repeatability (RSD%)	R^2
1	Benzene	5.328	0.67	0.9996
2	Toluene	6.769	0.63	0.9997
3	o-Xylene	8.063	0.58	0.9997
4	Ethyl benzene	8.760	0.60	0.9997
5	p-BFB	10.951	-	-
6	Tetralin	13.280	0.40	0.9996

■ Conclusion

An analytical study of alcohols, ketones, esters, ethers, and aromatics was carried out using the pre-configured sandwich-injection method of AOC-30's Sampler Navigator. Excellent results for both the linearity of the calibration curve and the repeatability of the peak area rations were obtained for all compounds, demonstrating the effectiveness of the function.

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01-00005-EN First Edition: Mar. 2021