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Application News

Analysis of Formaldehyde and Acetaldehyde in Ambient Air Using Integrated HPLC System

Miho Akagi

User Benefits

- Analysis of formaldehyde and acetaldehyde can be performed according to the Manual for measuring harmful air pollutants under air pollution control act.
- Highly robust analysis can be performed using integrated HPLC system i-Series LC-2070C and chemically durable analytical column Shim-pack Scepter C18-120.

■ Introduction

Aldehydes are emitted into the atmospheric environment through various production activities and automobile exhaust gas. Analytical methods and allowable levels for these aldehydes in ambient air have been established both domestically and internationally due to concerns about environmental pollution and human health impacts. Specifically for formaldehyde and acetaldehyde, analytical methods are described in Manual for Measuring Harmful Air Pollutants under Air Pollution Control Act[1] (described in only Japanese).

i-Series LC-2070C high-performance liquid chromatograph is suitable for routine analysis because of its compact design for integrating all HPLC units and easy maintenance. Shim-pack Scepter™ series HPLC columns are packed with a fully porous organosilica hybrid material, providing excellent durability and performance across a wide range of conditions.

This article presents an analysis of formaldehyde and acetaldehyde in ambient air using 2,4-dinitrophenylhydrazine (DNPH)-derivatization HPLC method with LC-2070C and Shim-pack Scepter C18-120. This method uses a DNPH cartridge to collect and derivatize aldehydes in ambient air. Subsequently, the aldehydes in the eluate from the cartridge are quantified and converted to concentrations in ambient air.

■ Analysis of Mixed Standard Solutions

DNPH-derivatized formaldehyde/acetaldehyde mixed standard solution (Fujifilm Wako Pure Chemical Corporation, P/N: 012-17391) was diluted with acetonitrile to seven concentrations (0.005, 0.01, 0.02, 0.05, 0.1, 0.2, 0.5 mg/L as formaldehyde and acetaldehyde) to prepare standard solutions for the calibration curve. These were subjected to HPLC analysis under the conditions shown in Table 1, and calibration curves were created using the peak areas of DNPH-derivatized formaldehyde and acetaldehyde in each standard solution. Fig. 1 shows the chromatogram of the mixed standard solution (0.5 mg/L), and Fig. 2 shows the calibration curve for formaldehyde and acetaldehyde. Good linearity was obtained for both components, with a coefficient of determination of 0.9999 or higher.

Table 1 Analytical Conditions

System	:	i-Series LC-2070C
Column	:	Shim-pack Scepter C18-120 (150 mm × 4.6 mm I.D., 3 µm)
Mobile phase	:	Water/Acetonitrile = 40 : 60
Flow rate	:	1.0 mL/min
Mixer	:	40 µL
Column temp.	:	30 ℃
Injection volume	:	10 µL
Vial	:	Shim-vial™ S, Amber glass *1
Detection (UV)	:	360 nm

*1 P/N : 227-34500-53

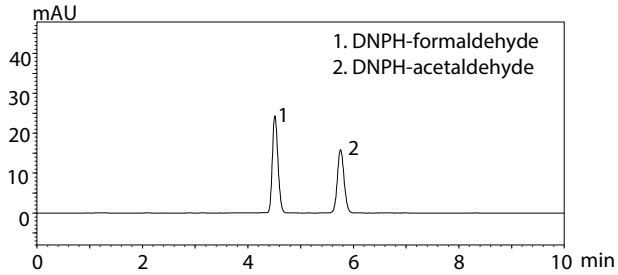

Fig.1 Chromatogram of mixed standard solution (0.5 mg/L)

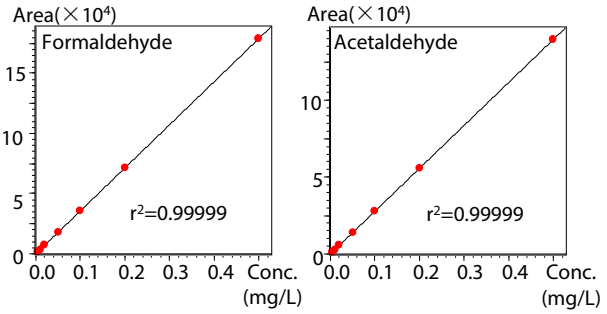

Fig.2 Calibration curves for formaldehyde and acetaldehyde

■ Repeatability

Six times consecutive analyses of mixed standard solution (0.005 mg/L) were performed. Obtained relative standard deviations (%RSD) for retention time and peak area are shown in Table 2 and Table 3. For both compounds even at low concentrations, relative standard deviations for retention time and peak area were within 0.2% and 1.6% respectively, demonstrating good repeatabilities.

Table 2 Retention time repeatabilities of mixed standard solutions (0.005 mg/L, n=6)

	Formaldehyde	Acetaldehyde
1	4.526	5.770
2	4.515	5.756
3	4.510	5.755
4	4.517	5.765
5	4.512	5.760
6	4.510	5.754
Average	4.515	5.760
%RSD	0.14	0.11

Table 3 Peak repeatabilities of mixed standard solutions (0.005 mg/L, n=6)

	Formaldehyde	Acetaldehyde
1	1716	1300
2	1707	1343
3	1709	1298
4	1663	1297
5	1744	1321
6	1692	1312
Average	1705	1312
%RSD	1.58	1.35

■ Analysis of Ambient Air Sample

Aldehydes in ambient air were collected at two outdoor locations (A and B) in Kyoto City following the Manual for measuring harmful air pollutants under air pollution control act. Two commercially available DNPH cartridges (GL Science Inc., P/N: 5010-23500) with an ozone scrubber (GL Sciences Inc., P/N: 5010-23510) connected upstream were used for collection. The cartridges were retrieved after aspirating air at 0.1 L/min for 24 hours using a pump mounted on an automatic gas sampler (Fig. 3). Sealed cartridges from the same lot as those for actual sampling were transported and handled identically for travel blank. Subsequently, these cartridges were eluted with acetonitrile, and the eluates were diluted to 5 mL to serve as the first-stage sample solution, second-stage sample solution, and travel blank sample solution, respectively (Fig. 4).

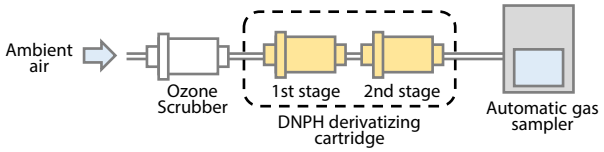

Fig.3 Outline of aldehyde collection and derivatization

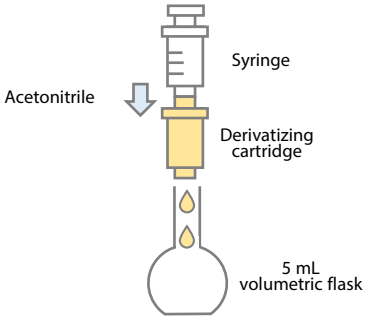

Fig.4 Outline of aldehyde elution procedure

Each sample solution was subjected to HPLC analysis under the conditions shown in Table 1. The resulting chromatograms are shown in Figs 5–10. DNPH-formaldehyde and DNPH-acetaldehyde were detected from the first-stage sample solutions collected at sampling sites A and B (Fig. 5 and Fig. 8). Additionally, trace amounts of DNPH-formaldehyde and DNPH-acetaldehyde were detected in the second-stage sample solutions and the travel blank sample solutions (Figs. 6, 7, 9, and 10).

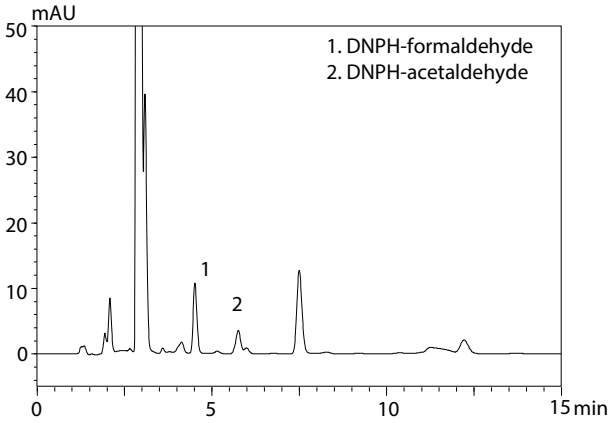

Fig. 5 Chromatogram of sample solution of site A・1st stage sample

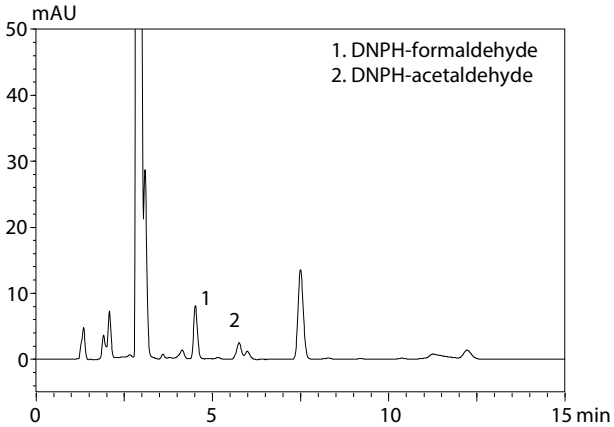

Fig. 8 Chromatogram of sample solution of site B・1st stage sample

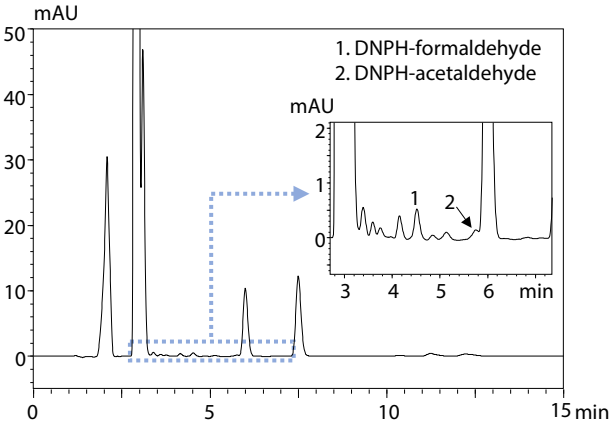

Fig. 6 Chromatogram of sample solution of site A・2nd stage sample
(Right: enlarged)

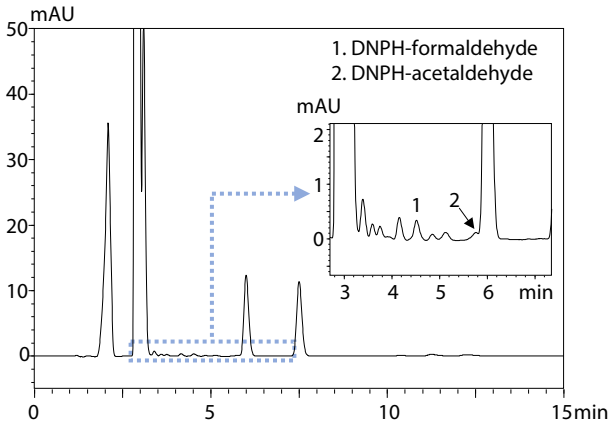

Fig. 9 Chromatogram of sample solution of site B・2nd stage sample
(Right: enlarged)

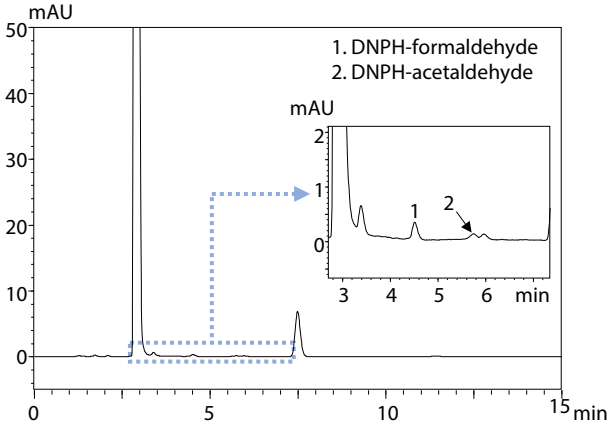

Fig. 7 Chromatogram of sample solution of site A travel blank
(Right: enlarged)

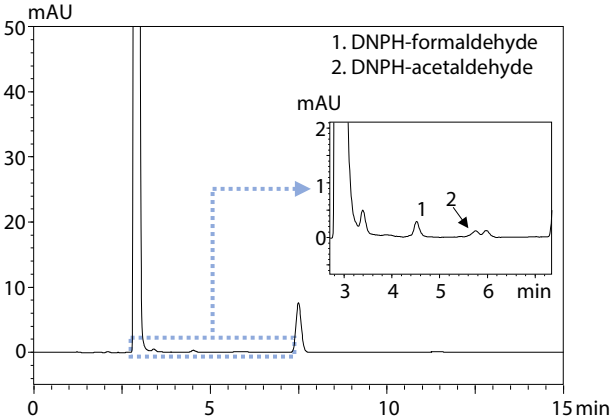

Fig. 10 Chromatogram of sample solution of site B travel blank
(Right: enlarged)

The formaldehyde and acetaldehyde concentrations in each sample solution were detected using respective peak areas and the calibration curve shown in Fig. 2. Table 4 shows the average concentrations from six consecutive analyses. It was concluded that sufficient aldehydes in the atmosphere were successfully captured since the first-stage sample solutions contained higher concentrations of aldehydes than those of the second-stage solutions.

Table 4 Obtained concentrations of formaldehyde and acetaldehyde in each sample (average)

Point	Test solution	Concentration of formaldehyde	Concentration of acetaldehyde
A	1st cartridge	0.220	0.132
	2nd cartridge	0.013	0.006
	Travel blank	0.007	<LOQ*
B	1st cartridge	0.165	0.090
	2nd cartridge	0.009	<LOQ*
	Travel blank	0.006	<LOQ*

*Under the limit of quantification

Furthermore, obtained average concentrations of formaldehyde and acetaldehyde in sample solutions were converted to concentrations in ambient air using formula 1. Sample solutions below the lower limit of quantification were calculated as 0 mg/L. The gas meter installed in the automatic gas sampler used in this investigation has a function to display the collected volume converted to 20 ℃ and 1 atm. The displayed collected volumes at site A and site B were 143.81 L and 144.06 L, respectively.

The conversion results are shown in Table 5. It was found that both locations A and B had several μg/m^3 of formaldehyde and acetaldehyde present in their ambient air.

$$C = \frac{(A_{S1} + A_{S2} - A_t) \times E \times 1000}{V} \quad \cdots \text{formula 1}$$

C : Each aldehyde concentration in ambient air at 20 ℃ [μg/m^3]
$As1$: Concentration of each aldehyde in 1st stage sample solution [mg/L]
$As2$: Concentration of each aldehyde in 2nd stage sample solution[mg/L]
At : Concentration of each aldehyde in travel blank sample solution [mg/L]
E : Volume of sample solution [mL]
V : Collected air volume measured with gas meter [L]

Table 5 Concentrations of formaldehyde and acetaldehyde in ambient air

Point	Formaldehyde	Acetaldehyde
A	7.9	4.8
B	5.8	3.1

■ Conclusion

Using i-Series LC-2070C the integrated HPLC system and Shim-pack Scepter C18-120 HPLC column, Quantitative analysis of ambient air by DNPH-derivatization HPLC method following the Manual for Measurement Methods of Harmful Air Pollutants under Air Pollution Control Act was performed. First, standard solutions were analyzed to confirm good linearity and repeatability even in low concentration range. Then using commercially available DNPH-cartridges to collect and derivatize aldehydes in ambient air, both aldehydes were retained on the column, separated, and detected with high sensitivity.

Relatively high concentrations of aldehydes around several μg/m^3 were detected because both two sampling sites were located close to heavy traffic roads. The method presented in this article is considered to be able to apply to the ambient air analysis at lower aldehyde levels because it has been confirmed to exhibit good linearity even in the low concentration range.

In addition, when a large number of interfering compounds are detected, the separations between aldehydes and co-eluted compounds would be improved by modifying the gradient profile and/or the column temperature.

＜Reference＞

1) Manual for Measuring Harmful Air Pollutants under Air Pollution Control Act. Part4, Capter1, “Method for measuring formaldehyde and acetaldehyde in ambient air”- Ministry of Environment, Government of Japan
https://www.env.go.jp/air/osen/manual2/index.html

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