Analysis of Formaldehyde in Air using Post-column Derivatization by High Performance Liquid Chromatography

Introduction

It is becoming a big concern that aldehydes such as the formaldehyde and acetaldehyde as an environmental pollutant may contaminate environment such as the atmosphere, lakes and marshes, reservoirs, and rivers. Therefore, it is an object of various regulations like Air Pollution Control Law, Water Supply Law, and Offensive Odor Control Law, etc. in this country. As a method to measure aldehydes in atmosphere using HPLC, general method employed is collecting aldehydes in atmosphere by using the sampler enclosing silicagel to contain 2,4- DNPH and extracting aldehydes derivatized after collection by the acetonitrile. JASCO has introduced so far the analysis of formaldehyde and acetaldehyde by the post column fluorescence derivatization method using 1,3-Cyclohexanedione as a labeling reagent.

Here, it was examined to use the same method to analyze aldehydes in atmosphere. As a result, aldehydes in atmosphere extracted by water were analyzed successfully as well as the case of aldehydes in water.

Keyword : Aldehyde genera, Passive gas tube, 1,3-Cyclohexanedione, Post column derivatization method, Shodex RSpak KC-811 6E, Fluorescence detector

Experimental

Equipment

Eluent pump:	PU-2080
Reagent pump:	PU-2085
Degasser:	DG-2080-53
Autosampler:	AS-2057
Column oven:	CO-2060
Reaction oven:	RO-2061
Detector:	FP-2020

Conditions

Column:	Shodex RSpak KC-811 6E (6.0 mmID x 250 mmL)
Eluent:	3 mM Perchloric acid
Flow rate:	1.0 mL/min
Reagent:	1,3-Cyclohexanedione in ammonium acetate buffer
Reagent flow rate:	0.4 mL/min
Column temp.:	60ºC
Reaction temp.:	120ºC
Wavelength:	Ex. 366 nm, Em. 440 nm, Gain x10
Injection volume:	50 µL
Standard sample:	Formaldehyde 0.1 mg/mL

Result

In Fig. 1, 1,3-Cyclohexanedione reaction formula of post column derivatization method is shown and Fig. 2 illustrates Flow system diagram.
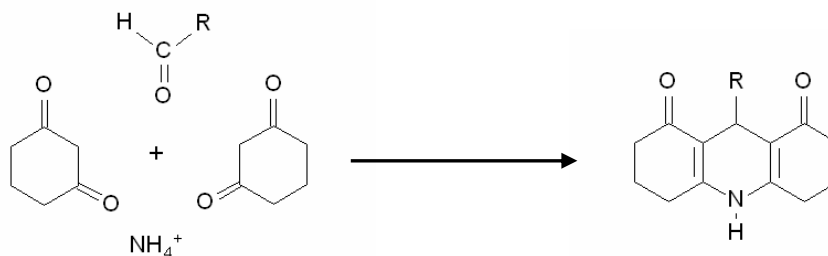
Decahydroacridine-1,8-dione

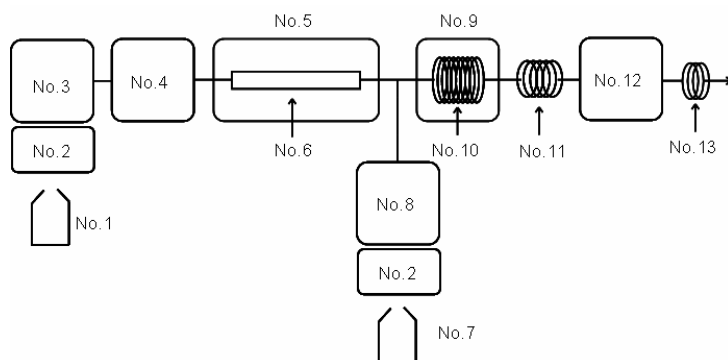**Fig. 1.** 1,3-Cyclohexanedione reaction formula of post column derivatization method

No.1 : Eluent
No.2 : Degasser (DG-2080-53)
No.3 : Pump for eluent (PU-2080)
No.4 : Autosampler (AS-2057)
No.5 : Column oven (CO-2060)
No.6 : Column (Shodex RSpak KC-811 6E)
No.7 : Reagent
No.8 : Pump for reagent (PU-2085)
No.9 : Reaction oven (RO-2061)
No.10: Reaction coil
No.11: Cooling coil
No.12: Fluorescence detector (FP-2020)
No.13: Backpressure coil

Fig. 2. Flow system diagram

Fig. 3 explains the usage of passive gas tube for collecting the sample in atmosphere and Fig. 4 shows the extraction method from passive gas tube.
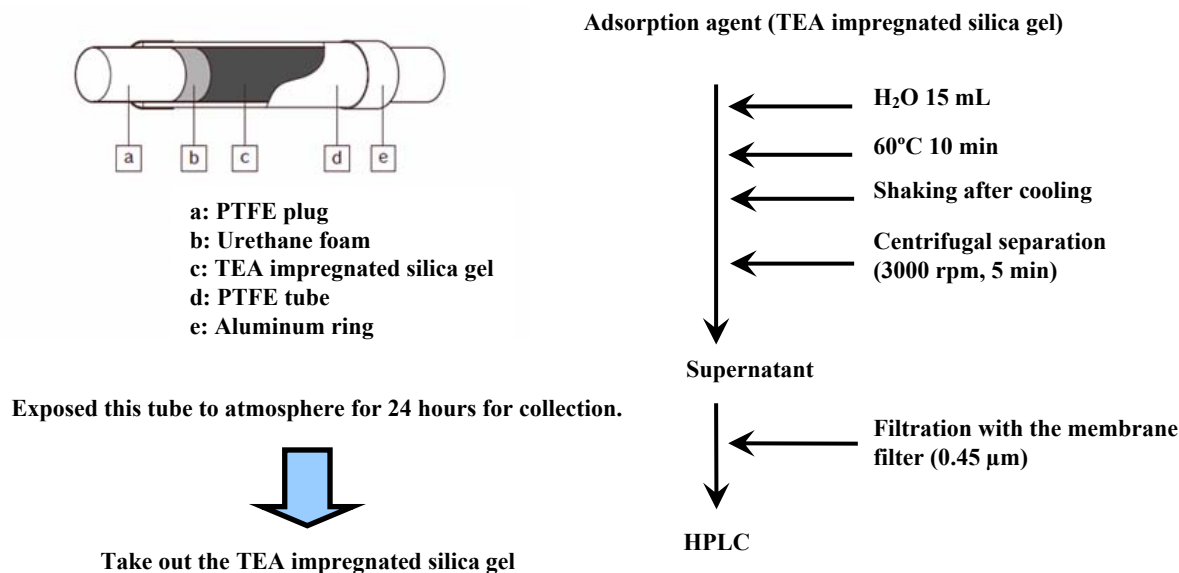
Adsorption agent (TEA impregnated silica gel)

H_2O 15 mL

60°C 10 min

Shaking after cooling

Centrifugal separation
(3000 rpm, 5 min)

Supernatant

Filtration with the membrane filter (0.45 µm)

HPLC

a: PTFE plug
b: Urethane foam
c: TEA impregnated silica gel
d: PTFE tube
e: Aluminum ring

Exposed this tube to atmosphere for 24 hours for collection.

Take out the TEA impregnated silica gel

Fig. 3. Usage of passive gas tube for collecting the sample in atmosphere

Fig. 4. Extraction method from passive gas tube

Fig. 5 shows the chromatogram of the sample in the laboratory atmosphere collected by passive gas tube. The calculated concentration of Formaldehyde obtained was 0.0054 mg/L.
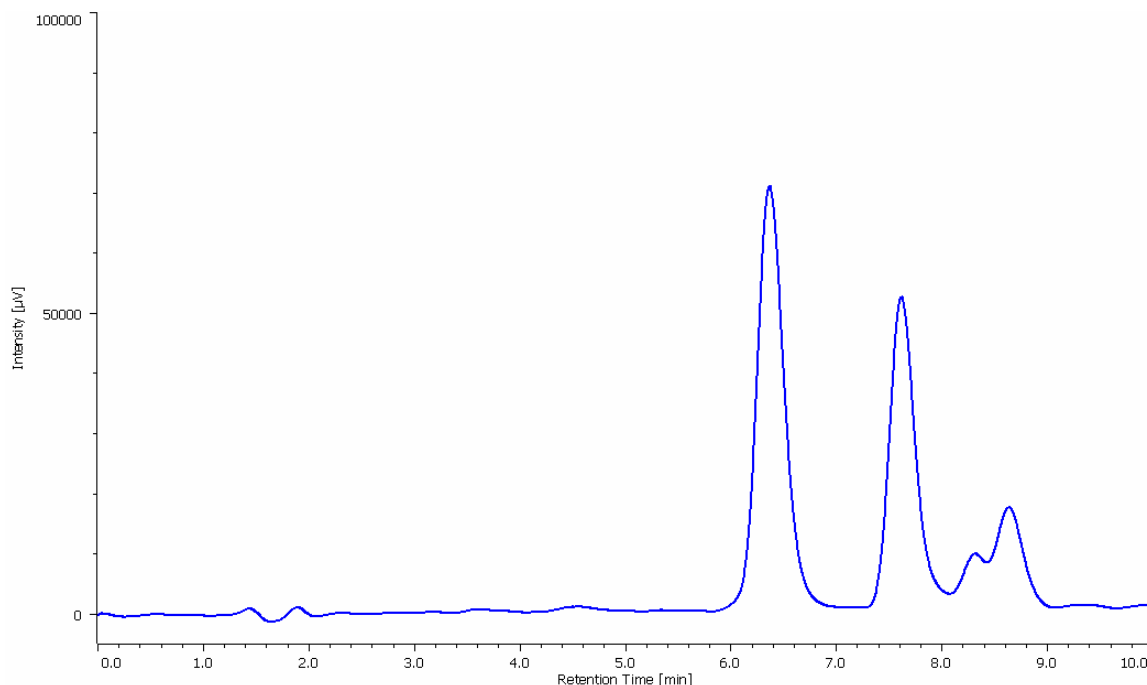

Fig. 5. Chromatogram of sample in the laboratory atmosphere
1: Formaldehyde (0.0054 mg/L)