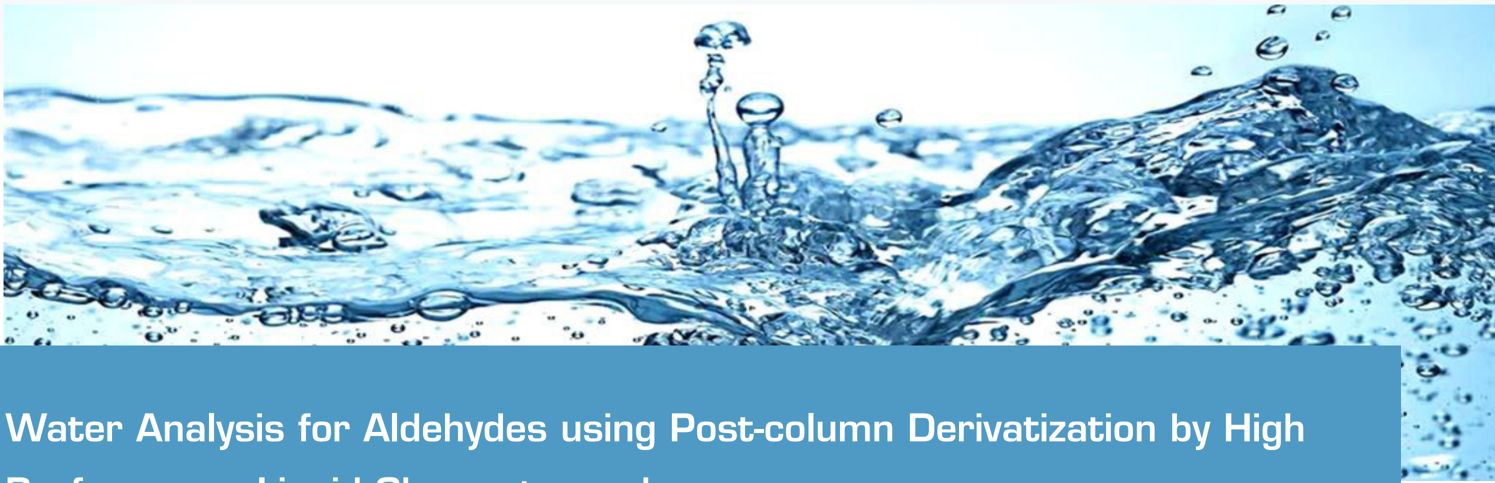
Application Note



Water Analysis for Aldehydes using Post-column Derivatization by High Performance Liquid Chromatography

Introduction

It is a growing concern that aldehydes such as formaldehyde and acetaldehyde may pollute air and water in lakes, reservoirs, and rivers in the environment. Therefore, the aldehydes are subjected to regulations by Air Pollution Control, Water Supply and Offensive Odor Control Acts in Japan.

Pre-column derivatization with 2,4-DNPH is known as a method to measure aldehydes in HPLC, however, the pretreatment of samples such as collection then condensation or extraction is required. JASCO introduced a method for the analysis of formaldehyde and acetaldehyde in water by post-column fluorescence derivatization using 1,3- Cyclohexanedione as derivatization reagent which did not require such pretreatment.

In this report, we extended the applicability of the method to other aldehydes by optimizing the derivatization conditions.

Keyword: Aldehydes, Formaldehyde, Acetaldehyde, 1,3-Cyclohexanedione, Post-column Derivatization, Shodex RSpak KC-8116E, Fluorescence detector

Jasco PU-2080-CO2

JASCO INC.
28600 Mary's Court, Easton, MD 21601 USA
Tel: (800) 333-5272, Fax: (410) 822-7526
Application Library: http://www.jascoinc.com/applications

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-8116E (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 L
Standard sample:	Formaldehyde, Acetaldehyde 0.1 mg/L each

Results

Figure 1 shows the chemical reaction of 1,3-Cyclohexanedione with aldehydes during the post-column derivatization and figure 2 shows flow path of the system.

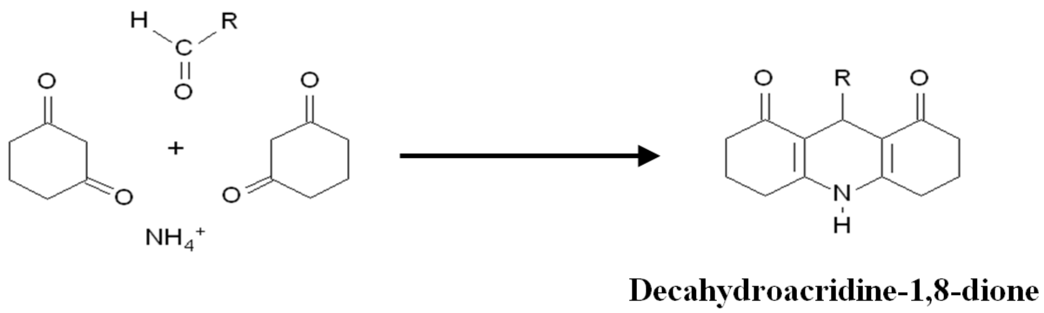


Figure 1. Chemical reaction of 1,3-Cyclohexanedione with aldehydes.

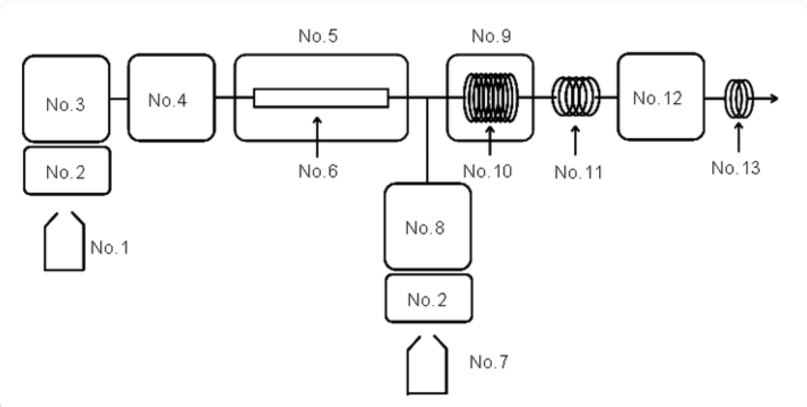


Figure 2. Flow line diagram of the system.

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

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Figure 3 shows chromatogram of the standard mixture of formaldehyde and acetaldehyde. The two components were separated within 8 minutes.

Figures 4, 5 and 6 show chromatograms of drinking water, river water and rain water, respectively. The top chromatograms are of neat samples and the bottom ones are of samples spiked by adding 0.1 mg/L of formaldehyde and acetaldehyde. All of the samples were separated.

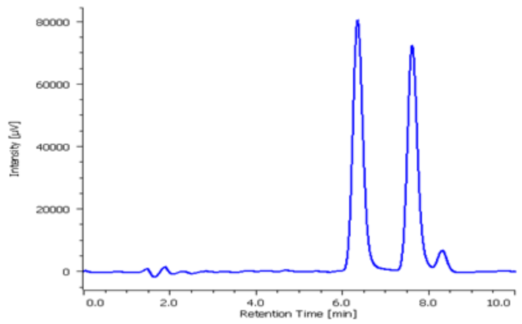

Figure 3. Chromatogram of the standard mixture of formaldehyde and acetaldehyde.
1: Formaldehyde, 2: Acetaldehyde (0.1 mg/L each)

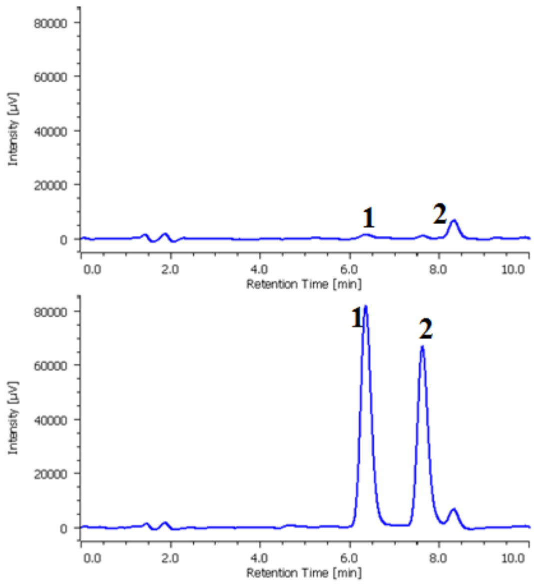

Figure 4. Chromatogram of drinking water.
1: Formaldehyde, 2: Acetaldehyde

Preparation. Drinking water was filtered using 0.45 μm membrane filter.

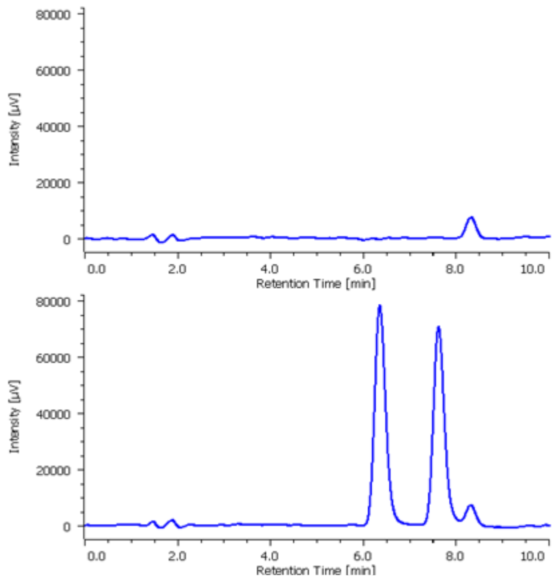

Figure 5. Chromatogram of river water.
1: Formaldehyde, 2: Acetaldehyde

Preparation. River water was filtered with 0.45 μm membrane filter.

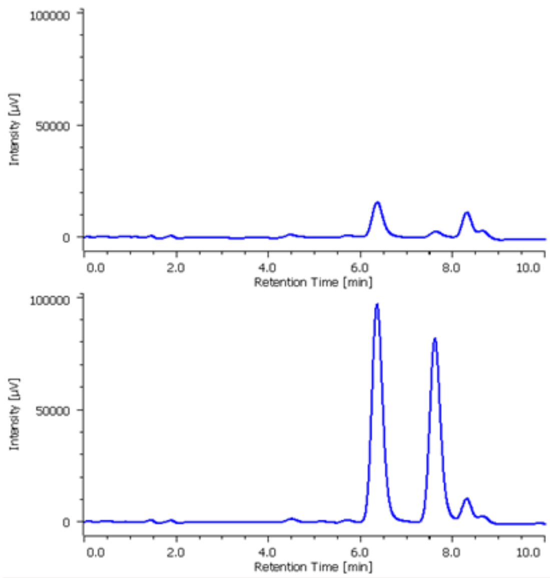

Figure 6. Chromatogram of rain water.
1: Formaldehyde, 2: Acetaldehyde

Preparation: Rain water was filtered with 0.45 μm membrane filter.

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