Application Note





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

Introduction

It is becoming a big concern that aldehydes such as the formaldehyde and acetaldehyde act as an environmental pollutant that may contaminate the atmosphere, lakes, marshes, reservoirs, and rivers. Therefore, aldehydes are subject to various regulations like the Air Pollution Control Law, Water Supply Law, and Offensive Odor Control Law in Japan. As a method to measure aldehydes using HPLC, the pre-column derivatization method by 2,4- DNPH is well known, but this requires pretreatment such as sample collection, condensation and extraction before analysis. JASCO has introduced the analysis of formaldehyde and acetaldehyde by the post column

luorescence derivatization method using 1,3- Cyclohexanedione as a derivatizing reagent which doesn't need such pretreatment.

The two above mentioned aldehydes and three others, propyl aldehyde, butyraldehyde, and valeraldehyde were analyzed simultaneously.

Keywords: Aldehydes, 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.:	60°C
Reaction temp.:	120°C
Wavelength:	Ex. 366 nm, Em. 440 nm, Gain x10
Injection volume:	100 µL
Standard sample:	Formaldehyde, Acetaldehyde, Propylaldehyde, Butylaldehyde, Valeraldehyde 0.1 mg/L each

Results

In figure 1, the 1,3-Cyclohexanedione reaction during the post column derivatization method is shown and figure 2 illustrates flow diagram for the HPLC.

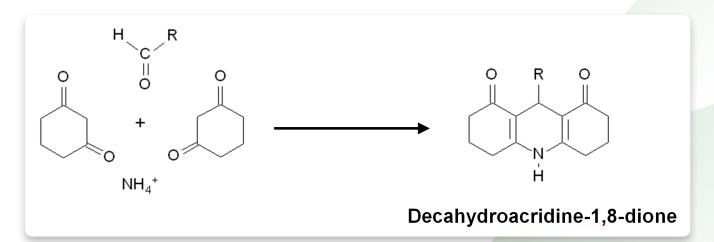


Figure 1. 1,3-Cyclohexanedione reaction formula of post column derivatization method



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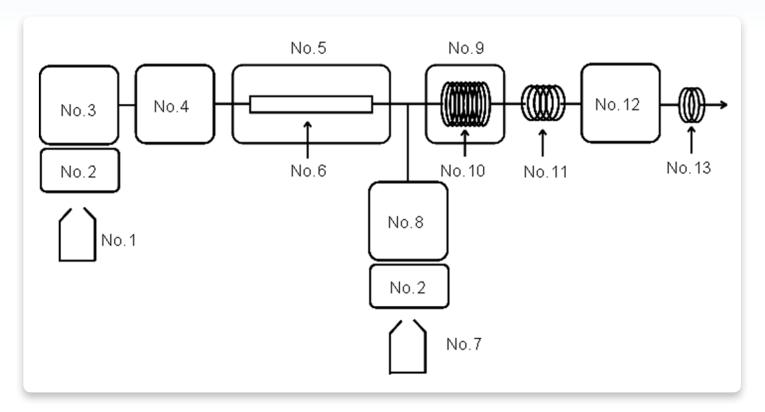


Figure 2. Flow system diagram

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



Figure 3 shows the chromatogram of 5 aldehydes. As shown, the 5 components were clearly separated within 16 min. The minimum detectable amount (in case of S/N=3) of each component is as below.

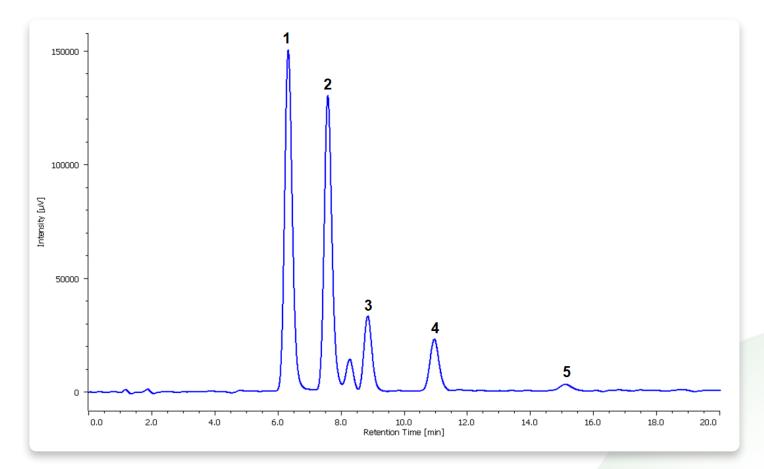


Figure 3. Chromatogram of 5 aldehydes and minimum detectable amount of each.1: Formaldehyde (0.091 ng), 2: Acetaldehyde (0.105 ng), 3: Propylaldehyde (0.418 ng), 4: Butylaldehyde (0.593 ng), 5: Valeraldehyde (4.53 ng)



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