Analysis of formaldehyde in clothing by HPLC with Pre-column Derivatization

In this note, a determination method for formaldehyde is described using a GL-7400 HPLC system.

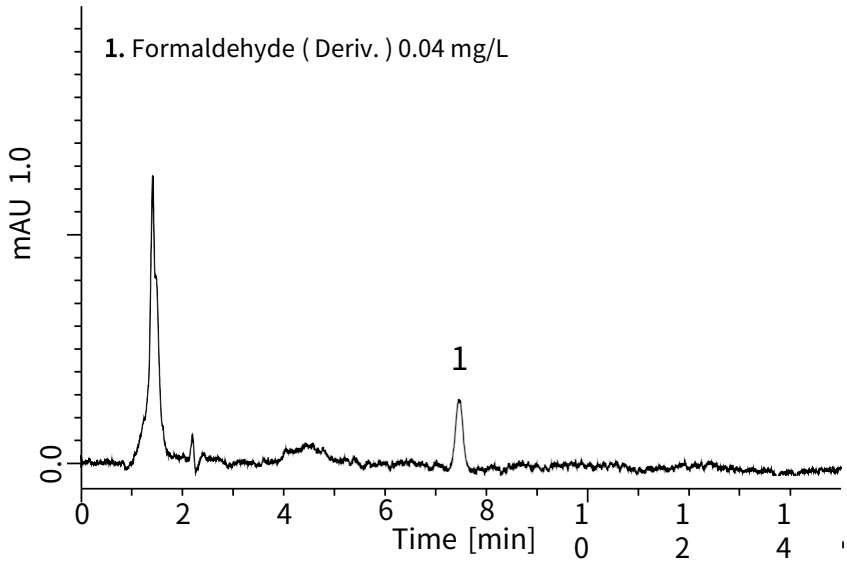
Formaldehyde is used as a component of various material, such as coating solution, adhesive agent, antiseptics, and resins. However, it is also a well-known chemical compound responsible for sick house syndrome. Formaldehyde released into indoor air can irritate eyes and mucous membranes. Furthermore, baby clothes containing formaldehyde cause severe skin inflammation.

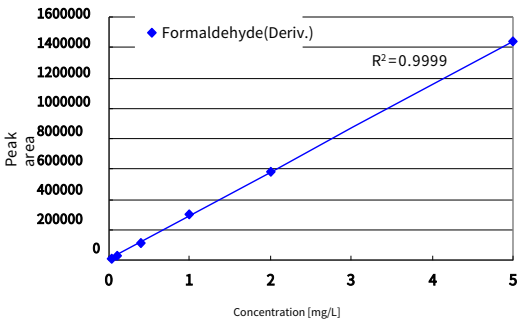
Therefore, formaldehyde contents of household products are regulated by law in Japan. The contents of baby clothes must be less than 16 ppm (=mg/kg), while those of other household products are required to be less than 75 ppm.

In this note, the determination of formaldehyde in clothing was performed compliant with the enforcement regulations of the law. The water extracts from samples were derivatized by acetylacetone (2,4-pentandione) and injected to the HPLC system coupled with UV-VIS detector.

A chromatogram obtained from standard solution

This is an example of an analysis of derivatized formaldehyde.The concentration described below is the concentration of the solution before derivatization. In case the pretreatment method in the next page is applied to a sample, the concentration used in this chromatogram 0.04 mg/L can be converted to 1.6 mg/kg.





The calibration curve of formaldehyde

* HPLC analysis was performed after the derivatization reaction of formaldehyde. The standard solution was gradually diluted and each were derivatized then analyzed. The concentration in the graph is the concentration of diluted solution before derivatizing using acetylacetone solution.

Conditions

Column:	Inertsil ODS-3 (4 µm, 150 x 4.6 mm I.D.)
Column Cat. No. :	5020-04645
Eluent:	A) CH_3CN B) H_2O A / B = 15 / 85, v/v (gradient mixer)
Flow Rate:	1.0 mL/min
Col. Temp.:	40 °C
Detection:	VIS 413 nm
Injection vol.:	10 µL

sample pretreatment

Sample
- 2.5g

Extraction
- Water 100 mL
- Place in a water bath at 40°C for 1hour and shake a few times
- Filtration while warm

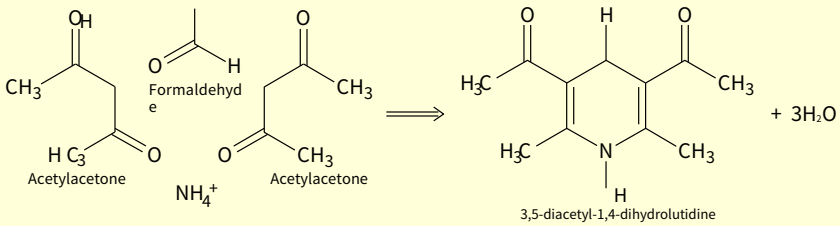
Derivatization
- Extract 5 mL
- 5mL Acetylacetone solution*1
- Place in a water bath at 40 °C for 30min
- Wait for 30 min

HPLC-UV

*1 acetylacetone solution:
To 150 g of ammonium acetate, 3 mL of acetic acid and , 2 mL of acetylacetone were added. The solution was diluted with water and made up to 1 L.

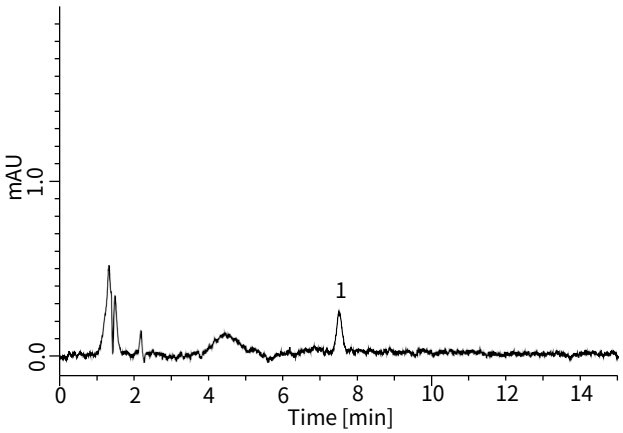
Derivatization reaction

One molecule of formaldehyde reacts with two molecules of acetylacetone and one molecule of ammonium ion. As a result of the reaction, 3,5-diacetyl-1,4-dihydrolutidine, which can be sensitively detected by UV-Vis detector, is generated.

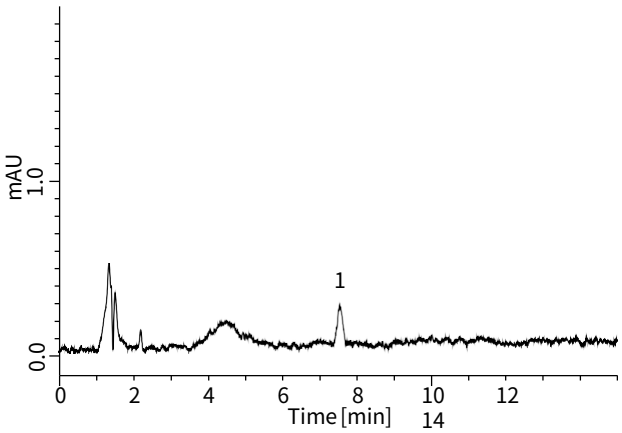


Structures are created using Chemistry 4-D Draw which is provided by ChemInnovayion Software, Inc.

Polyester fiber extract



Cotton extract



1. Formaldehyde (Deriv.)

GL Sciences Inc. Japan
22-1 Nishishinjuku 6-chome
Shinjuku-ku, Tokyo
163-1130, Japan

Phone: +81-3-5323-6620
Fax: +81-3-5323-6621
Email: world@gls.co.jp
Web: www.glsciences.com

GL Sciences Inc. USA
4733 Torrance Blvd. Suite 255
Torrance, CA 90503
USA

Phone: +1-310-265-4424
Fax: +1-310-265-4425
Email: info@glsciencesinc.com
Web: www.glsciencesinc.com

GL Sciences B.V.
Dillenburgstraat 7C
5652AM, Eindhoven
The Netherlands

Phone: +31-40-254-9531
Email: info@glsciences.eu
Web: www.glsciences.eu

GL Sciences (Shanghai) Limited
Tower B, Room 2003
Far East International Plaza
No.317 Xianxia Road, Changning District
Shanghai, China 200051

Phone: +86-21-62782272
Email: contact@glsciences.com.cn
Web: www.glsciences.com.cn

GL Sciences

International Distributors
Visit our Website at www.glsciences.com/distributors