

Application News

Gas Chromatography

No.G271A

Analysis of Vinylidene Chloride in Polyvinylidene Chloride Plastics by GC

Residual organic solvents in food packaging materials are receiving attention due to the heightened concern for food safety and public health. Specific standards and specifications as well as testing methods are established for each type of material used in food packaging materials in Japan's "Food Sanitation Act-Section 3: Implements, Containers, and Packaging in the Standards and Criteria for Food and Food Additives, etc."

Polyvinylidene chloride is a polymer that is transparent, displays excellent water resistance, chemical resistance, and gas-barrier properties, and is resistant to temperatures in the range of 140 to

170 °C. In addition to household wrapping film, it is used as a film for wrapping foods that have been heated to high temperatures.

Vinylidene chloride is the monomer of polyvinylidene chloride, and long-term oral exposure is said to adversely affect the liver and kidney. Due to the possibility that vinylidene chloride may persist in products, polyvinylidene chloride material testing has been established as a separate standard in the Food Sanitation Act.

This Application News introduces an example of analysis of vinylidene chloride in plastic with polyvinylidene chloride as its principal ingredient.

■ Analytical Method

The vinylidene chloride test method is for measuring vinylidene chloride monomer present in polyvinylidene chloride by GC/FID using the headspace method.

Sample pretreatment is conducted according to that specified in the "Food Sanitation Act-Section 3: Implements, Containers, and Packaging in the Standards and Criteria for Food and Food Additives, etc." Commercially-available polyvinylidene chloride household wrapping film was used as the sample, and the vinylidene chloride in the sample was analyzed.

A porous polymer PLOT (porous layer open tubular) CP-PoraBOND Q column was used. The carrier gas flowrate was set so that the vinylidene chloride would elute at about 9 minutes. The analytical conditions are shown in Table 1.

Following is an overview of the analytical procedure.

(1) Test Solution Preparation

Cut and weigh out a 0.5 g fragment of sample, and transfer it to a headspace vial. Next, add 2.5 mL *N,N*-dimethylacetamide, seal the vial, and use this as the test solution.

(2) Standard Solution Preparation

Transfer 50 μ L vinylidene chloride standard solution (60 μ g/mL) to a vial containing 2.5 mL *N,N*-dimethylacetamide, and seal the vial. Use this as the standard solution.

(3) Measurement

Heat the sealed vials of test solution and standard solution for 1 hour at 90 °C, and introduce 0.5 mL of the respective gas phases into the gas chromatograph. For the gas chromatograph column, use a 3 μ m styrene-divinylbenzene porous polymer-coated column, and conduct analysis by GC/FID.

(4) Determination

Compare the detection times of test solution peak and the vinylidene chloride standard solution peak. If the retention times correspond, compare their respective peak areas. Verify that the test solution vinylidene chloride peak area is not greater than that of the vinylidene chloride standard solution peak area (6 μ g/g or less in the material).

■ Analysis of Standard Solution and Sample Solution

The chromatograms of the vinylidene chloride standard solution and the test solution prepared from commercially-available household wrapping film, of which the principle ingredient is polyvinylidene chloride, are shown in Fig. 1.

The vinylidene chloride peak in the test solution chromatogram has a smaller peak area than that of the standard solution, confirming that it is smaller than the reference value.

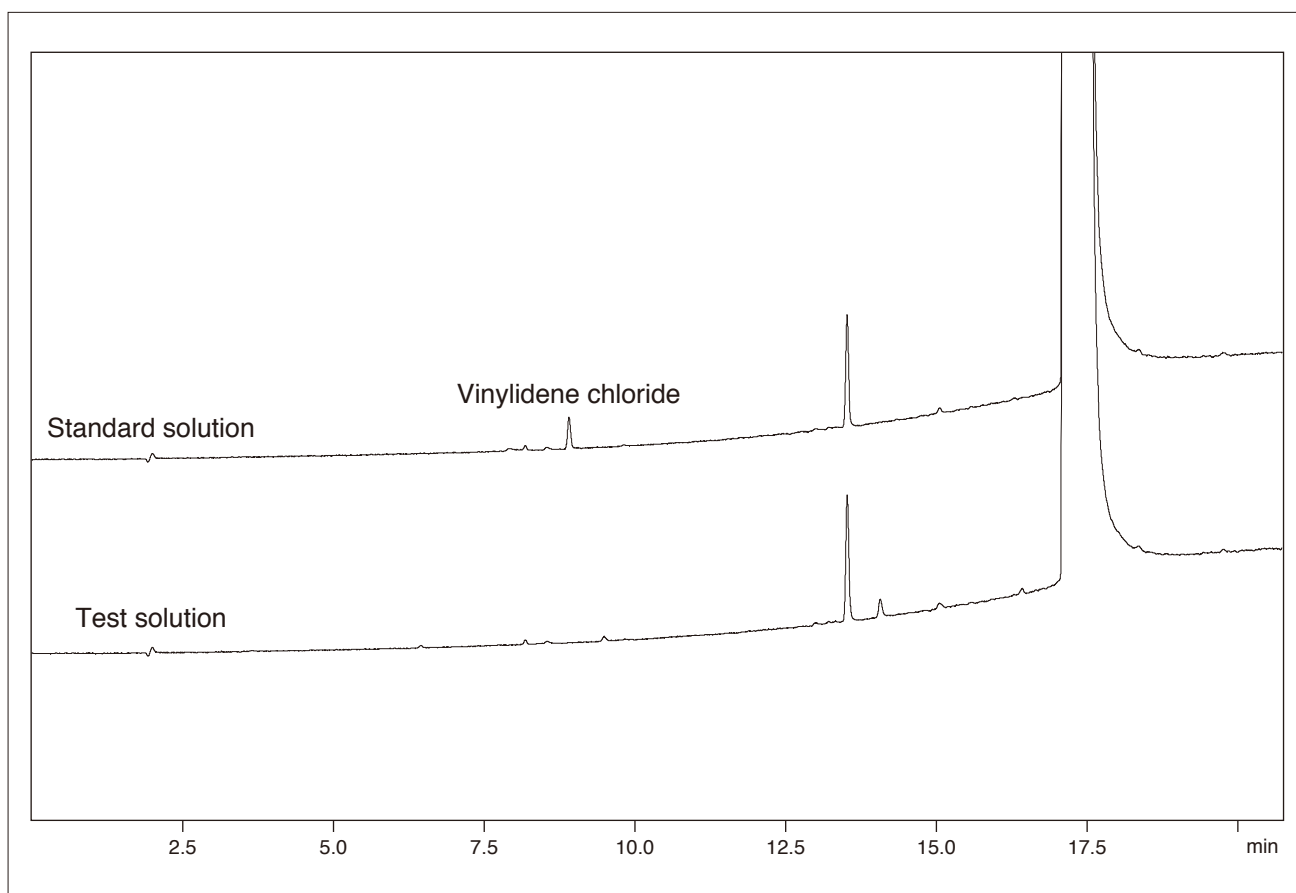


Fig. 1 Chromatograms of Vinylidene Chloride Standard Solution and Test Solution

Table 1 Analytical Conditions

Model	: TurboMatrix HS-40 + GC-2010PlusAF
Column	: CP-PoraBOND Q FUSED SILICA (25 mL, × 0.25 mmI.D. df = 3 μm)
Column Temperature	: 80 °C (1 min)-10 °C/min-250 °C (10 min)
Injection Temperature	: 200 °C
Carrier Gas	: He 30 cm/sec
Detector	: FID
Detector Temperature	: 250 °C
Injection Volume	: 0.5 mL
Sample Thermostatting	: 90 °C, 60 min

[References]

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Food Sanitation Act – Section 3: Implements, Containers, and Packaging in the Standards and Criteria for Food and Food Additives, etc.



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