

Application News

Gas Chromatography

No.G276

Analysis of Vinyl Chloride Leached from Metal Food Can by GC

Heightened concern for food safety and public health has brought greater attention to residual organic solvents in food packaging materials.

Individual standards and specifications as well as testing methods were established for each type of material used in these food packaging materials in Japan's "Food Sanitation Act-Standards and Criteria for Food and Food Additives, etc. Chapter III Apparatus and Containers and Packaging."

It is common practice for metal cans used as food containers to be coated internally with synthetic resin materials consisting of epoxy resin, phenolic resin,

polyvinyl chloride (PVC), etc. These materials then come into direct contact with the food contained therein.

Internally coated cans are subject to a separate standard which requires that they undergo leachate testing as the established test method for phenol, formaldehyde, evaporation residue, epichlorohydrin, and vinyl chloride.

This Application News introduces an example of analysis for leached vinyl chloride from the synthetic resin coating obtained from the vinyl liner of a metal can used for foods.

■ Overview of Vinyl Chloride Analysis Method

The test method described here is a headspace GC-FID method for measuring vinyl chloride leached from the can's internal coating into a leaching solution, consisting of ethanol cooled to 5 °C or less.

The contents, consisting of food in either in liquid, gel or solid state, contact the interior surface of the can material. Hence, metallic cans that employ synthetic resin coatings that contact the food directly are the target of measurement in this case.

Cans that are manufactured for the purpose of long-term preservation of food are routinely subjected to

some type of bacteria removal or sterilization. However, regardless of the type of food or method of sterilization, they are all subject to testing as described here. (Note: Cans that are used for dried food or solid food that make little contact with the interior surface - except for oily and fatty foods - are not subject to testing.)

In order to pass this test, the peak area of the vinyl chloride dissolved in the ethanol leachate must not exceed the peak area of vinyl chloride standard solution (0.05 µg/mL).

■ Analytical Method

Sample pretreatment was conducted in accordance with the above-mentioned "Food Sanitation Act-Standards and Criteria for Food and Food Additives, etc. Chapter III Apparatus and Containers and Packaging", as described below.

(1) Preparation of Test Solution

The sample was a commercially-available food can with an interior lining. The sample can was filled with cold ethanol at less than 5 °C, and leaching was allowed to take place over a period of 24 hours at below 5 °C. This leachate was then analyzed by gas chromatography (GC): 10 mL of leachate was transferred to a headspace vial, which was then immediately sealed.

(2) Preparation of Standard Solution

50 µL of vinyl chloride standard solution (10 µg/mL), cooled prior to analysis using methanol and dry ice, was transferred to a headspace vial already containing 10 mL of ethanol, after which the vial was immediately sealed.

(3) Measurement

The sealed vials of leachate and standard solution were heated at 50 °C for 30 minutes. Their respective headspace gases were then injected into the GC for measurement. The column used was 0.25 mm I.D. CP-PoraBONDQ. The carrier flowrate was adjusted so that vinyl chloride would elute in approximately 5 minutes. Measurement was conducted utilizing the

split injection method, with the split ratio being optimized for best peak shape and sensitivity.

The preparation procedure is shown in Fig. 1 and the analytical conditions are shown in Table 1.

(4) Assessment

The elution times of the leachate peak and vinyl chloride standard solution peak are compared. If they coincide, then their peak area values are compared. In order to pass the test, the area of the vinyl chloride peak (if any) in the sample leachate should not exceed the vinyl chloride peak area of the standard solution. (Vinyl chloride: 0.05 µg/mL maximum)

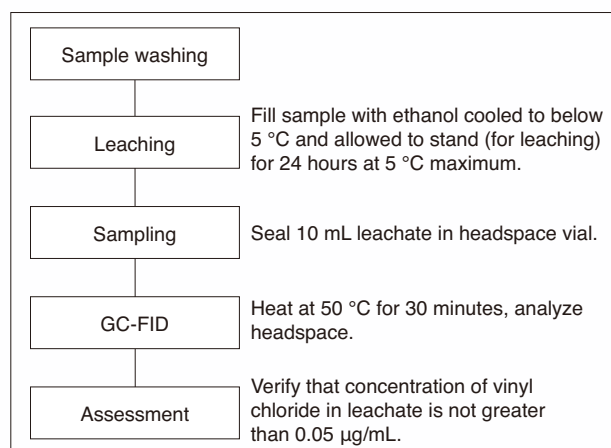


Fig. 1 Preparation of Metal Food Can

■ Analysis of Standard Solution and Test Solution

Fig. 2 shows chromatograms resulting from analysis of a vinyl chloride standard solution and leachate from a commercially-available lined metallic food can. Vinyl

chloride was not detected in the leachate from the sample can. (Fig. 2)

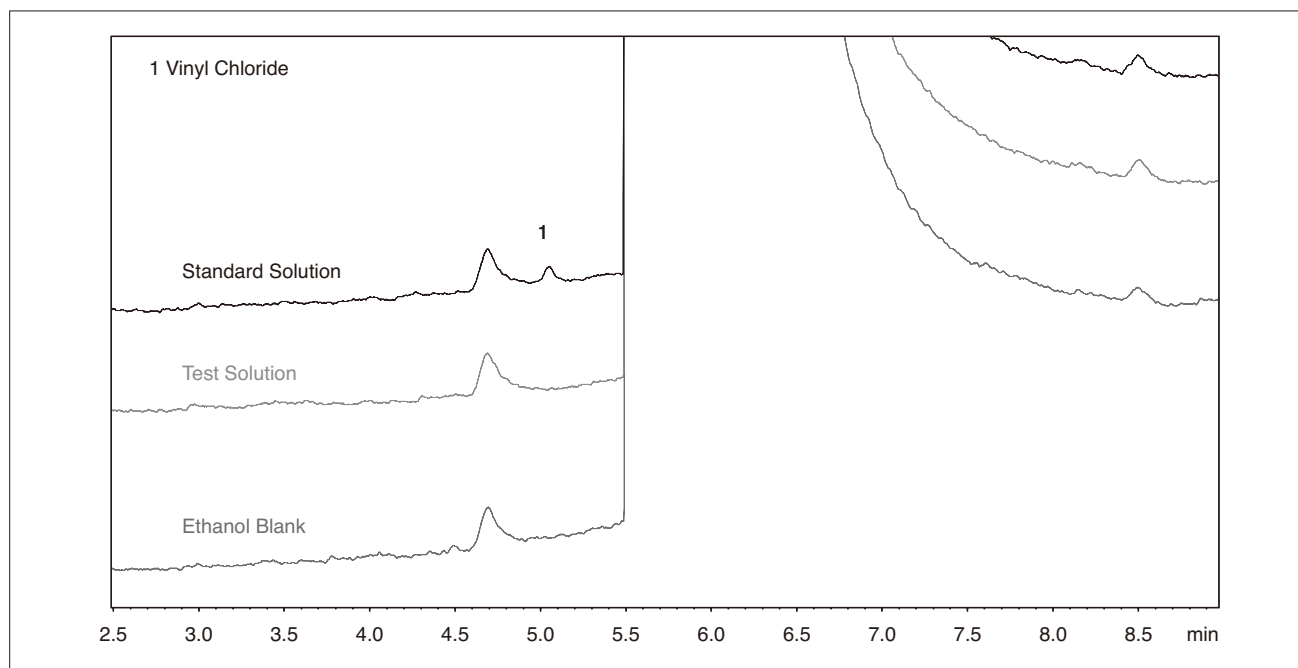


Fig. 2 Chromatograms of Standard Solution and Test Solution

Table 1 Analytical Conditions

Model	: TurboMatrix HS-40 + GC-2010 Plus AF ¹⁾
Column	: CP-PoraBOND Q FUSED SILICA (25 m × 0.25 mmI.D. df = 3 μm)
Column Temp.	: 80 °C (1 min)- 10 °C/min - 250 °C (10 min)
Injection Temp.	: 200 °C
Carrier Gas	: He 83.5 kPa (0.95 mL/min at 80 °C)
Detector	: FID ²⁾
Detector Temp.	: 250 °C
Injection Volume	: 0.5 mL (Injection Time: 0.2 min, Split 1:2.6)
Sample Thermostatting	: 50 °C, 30 min

1) A 0.18-mm inside diameter open tube was used for the HS-40 transfer line.

2) As this test constitutes trace analysis, high-purity air was used for measurement.
Sensitivity may be affected by the instrument condition and utility.

[References]

Japan's Ministry of Health, Labour and Welfare Notification No. 201, March 31, 2006

"Food Sanitation Act-Standards and Criteria for Food and Food Additives, etc. Chapter III Apparatus and Containers and Packaging"



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