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Application News

No.G277

Gas Chromatography

Analysis of Vinyl Chloride in Polyvinyl Chloride Plastics by Headspace Gas Chromatography with a Flame Ionization Detector (GC/FID)

The 16th revision of the Japanese Pharmacopeia was announced in the Japanese Ministry of Health, Labour and Welfare Notification No. 65 dated March 24, 2011, and its use became effective as of April 1, 2011. In this revision, under the Test Methods for Plastic Containers, the headspace GC/FID method has been adopted as the test method for vinyl chloride in polyvinyl chloride injection containers used for aqueous solutions.
This Application News describes the test method for analysis of vinyl chloride contaminants in polyvinyl chloride injection containers used for aqueous solutions as specified in the 16th revision of the Japanese Pharmacopeia.

System Suitability Test

Transfer 2.5 mL *N,N*-dimethylacetamide to a headspace vial, followed by 50 µL vinyl chloride standard solution (10 µg/mL in ethanol, previously chilled using methanol and dry ice). Seal the vial and use this as the standard solution.
Analyze the standard solution using the conditions specified in Table 1, and verify the resolution and repeatability.
System suitability test for chromatographic resolution:
The chromatographic resolution criteria of vinyl chloride and ethanol is specified as R = 3.0 or greater.
The chromatogram obtained from analysis of the standard solution is shown in Fig. 1. Resolution greater than 3.0 was obtained.

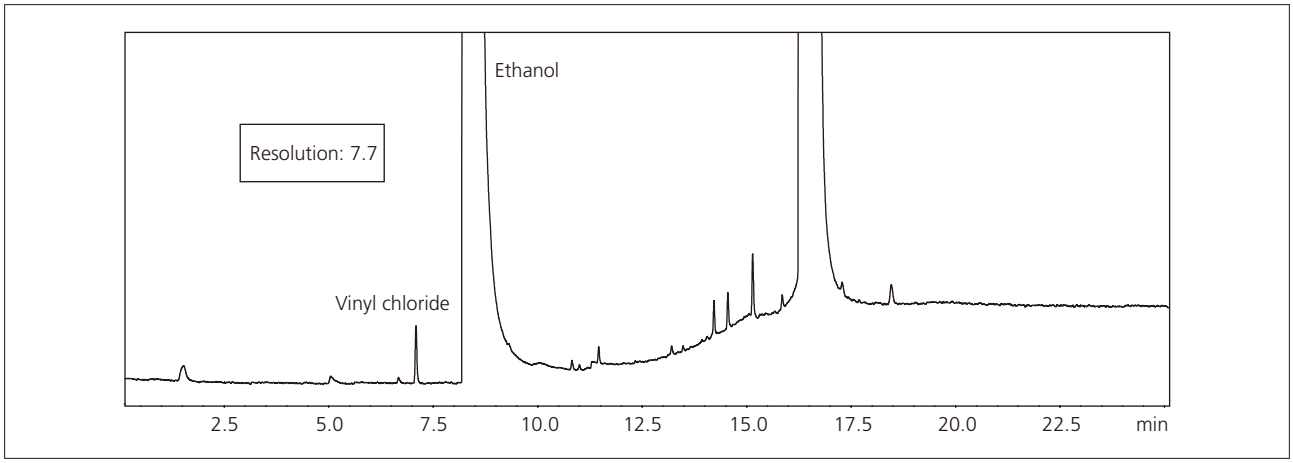

Fig. 1 Chromatogram of Suitability Test for Chromatographic Resolution

Table 1 Analytical Conditions

Model	: TruboMatrix HS-40 (short transfer) + GC-2010 Plus AF
Column	: CP-PoraBOND Q FUSED SILICA (25 m × 0.25 mmI.D. df = 3 µm)
Column Temp.	: 50 °C (2 min) → 10 °C/min → 120 °C (10 min) → 20 °C/min → 250 °C (10 min)
Injection Temp.	: 200 °C
Carrier Gas	: He 1.58 mL/min at 50 °C
Detector	: FID
Detector Temp.	: 250 °C
Injection Volume	: 0.5 mL
Sample Thermostatting	: 90 °C, 60 min
HS Pressuer	: 135 kPa (transfer tube 0.25 mmI.D.)
Split Ratio	: 1:5

System Repeatability Check (Peak Area):
The specification requires that the relative standard deviation (RSD) of peak areas obtained from 6 consecutive analyses of a vinyl chloride standard solution be 5.0 % or less.

Fig. 2 shows the magnified chromatograms obtained from 6 consecutive analyses of the standard solution, and the peak area repeatability is shown in Table 2.
The relative standard deviation was 2.3 %, and met the 5.0 % repeatability criteria.

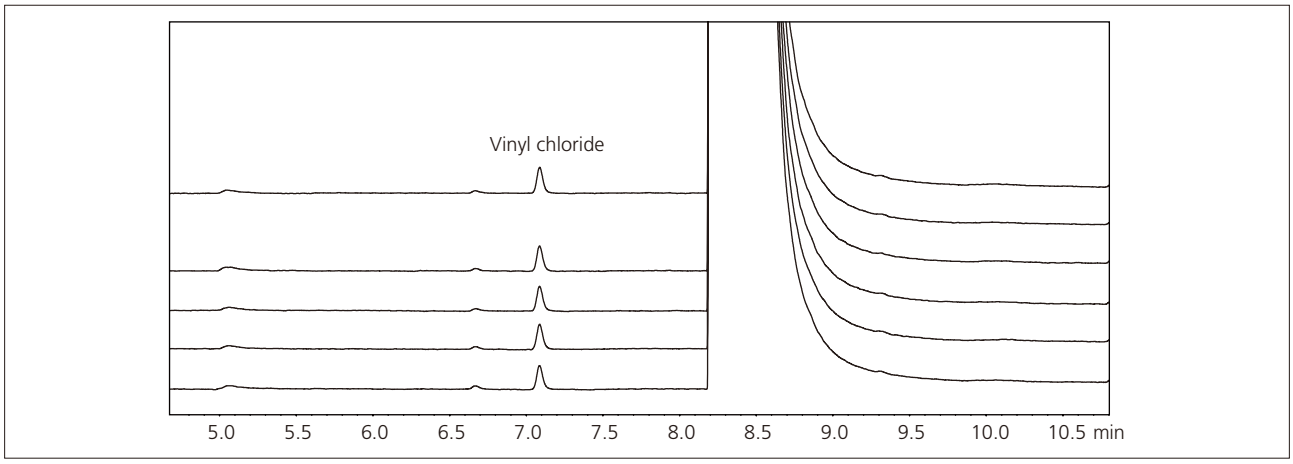

Fig. 2 Chromatograms Showing Repeatability of Vinyl Chloride

Table 2 Repeatability of Vinyl Chloride Peak Area

	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	Average Value	Relative Standard Deviation (%)
Vinyl Chroride Peak Area	1479	1419	1457	1444	1448	1382	1438	2.3

*The area values and relative standard deviation are reference values, and are not guaranteed values.
*Due to the large influence that preparation of the standard solution has on repeatability, care must be taken in sealing of the vial.

Analysis of a Sample Solution

Prepare the sample by first washing the polyvinyl chloride material with water and wiping it thoroughly with filter paper. Then cut off a piece of sample no larger than 5 mm^2, transfer a 0.5 g fragment of the sample to a headspace vial, add 2.5 mL *N,N*-dimethylacetamide, seal the vial, and use this as the test solution. If it is difficult to dissolve the sample, allow it to stand overnight at ambient temperature.

Analyze the standard solution and sample solution by headspace GC/FID, and verify that the vinyl chloride peak area in the sample solution chromatogram has a smaller peak area than that in the standard solution. The standard solution and sample solution chromatograms are overlaid for comparison in Fig. 3.
Vinyl chloride was not detected in the sample solution used in this analysis.

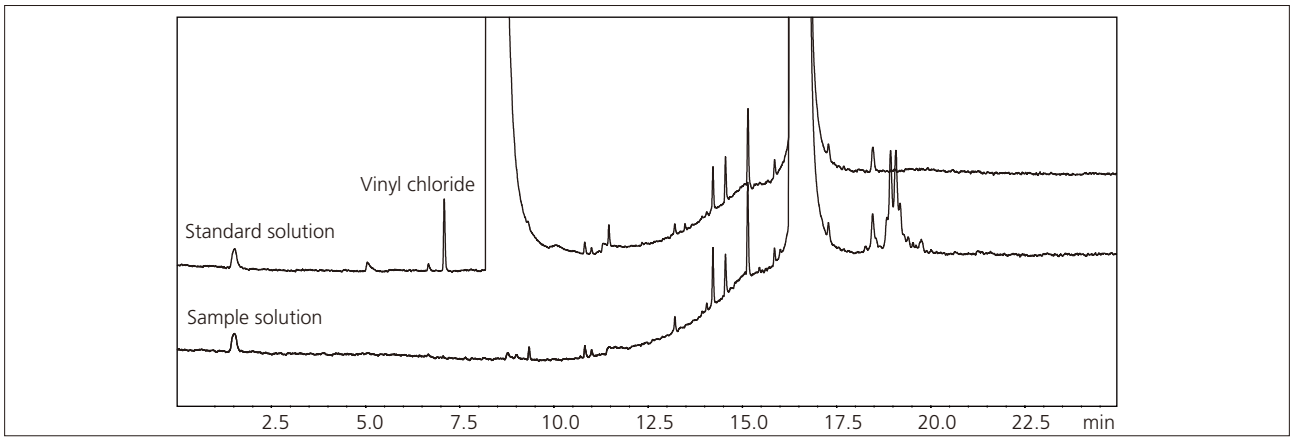

Fig. 3 Chromatograms of Standard Solution and Sample Solution

For details regarding this test method, please refer to the Japanese Ministry of Health, Labour and Welfare Notification No. 65 (March 24, 2011).
*As high sensitivity is required for analysis of trace level components in this test, use of a high purity air cylinder or an air purification system is recommended.

[References]
The Japanese Ministry of Health, Labour and Welfare Notification No. 65 (March 24, 2011)
16th Revision of the Japanese Pharmacopoeia

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