LAAN-A-GC-E030

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Application News

Gas Chromatography No.G264

Fast Analysis of Acrylonitrile in ABS Polymer Using a Backflush GC System

Residual organic solvent in food packaging materials is a problem that receives serious attention because of the implications to food safety and public assurance. While food containers and packaging materials are subject to standards controlled through material testing and testing of leachates from the packaging materials, as specified in Japan's Food Sanitation Law, the recent heightened concern over safety and assurance has led to the analysis of substances other than those subject to the current control standards in Japan. One example of these is the measurement of acrylonitrile, which is controlled as a carcinogen by the U.S. Food and Drug Administration (FDA) and by regulations in the European Union (EU).
Flame ionization detectors (FID) and flame thermionic detectors (FTD) are used for GC measurement of acrylonitrile, but this analysis is often time consuming due to the presence of high-boiling substances in the liquid extracted from resinous materials, which are slow to be driven from the column.
In addition, when an FTD is used as the detector, reduced stability and deterioration of the detector are concerns, depending on the solvent that is used.
A backflush GC system is one in which the carrier gas flow is reversed after the elution of the analytes from the column, so that the high-boiling substances are expelled from the injector split vent. This elimination of the backflushed high-boiling substances not only serves to protect the column and prevent contamination of the detector, it can greatly shorten the analysis time.
This Application News introduces a fast analysis of acrylonitrile based on the use of the backflush GC system.

■ Backflush GC System

In a backflush GC system, a special backflush element, consisting of an advanced pressure controller (APC) and a connection fitting, is connected at the separation column outlet. The backflush element allows control of the column outlet pressure so that flow through the column can be reversed.
When backflushing is conducted, the pressure in the backflush element is raised following detection of the analytes while the pressure in the sample injection unit is simultaneously lowered, thereby reversing the pressure gradient in effect during usual analysis. As a result, the carrier gas flow is reversed, and residual contaminants in the column are expelled via the injection unit split vent. This technique shortens the analysis time, protects the column, and prevents contamination of the detector.

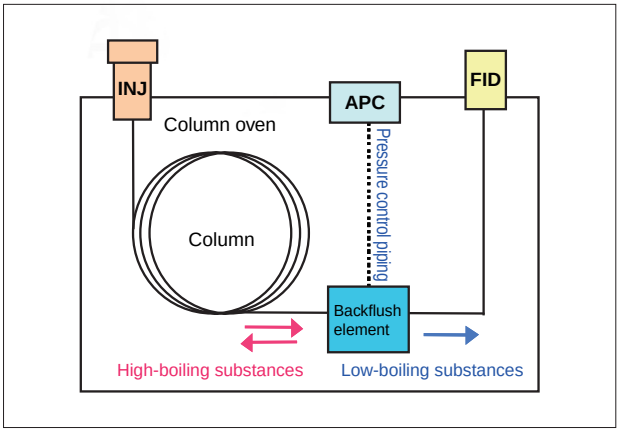

Fig. 1 Backflush GC System

■ Analysis Method (ABS Resin Pretreatment)

Pretreatment of the ABS sample was conducted according to "Food Sanitation Inspection Guidelines, Physics and Chemistry, 2005".
The test solution was prepared by cutting a commercially available spoon made of the ABS resin into fragments 5 mm square, and dissolving them in N,N-dimethylformamide. An acrylonitrile standard sample was added to this blank test solution to prepare a standard addition sample.
An Rtx-WAX column (30 m 0.25 mm I.D. 0.25 µm) was used for the analysis, and the standard-addition sample was analyzed by GC/FID and GC/FTD.
The sample preparation flow diagram is shown in Fig. 2, and the analytical conditions are shown in Table 1.

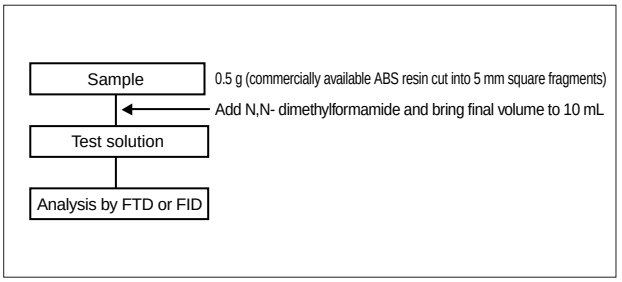

Fig. 2 Preparation of ABS Polymer Sample (according to Food Sanitation Inspection Guidelines, Physics and Chemistry, 2005)

Table 1 Analytical Conditions

Column	: Rtx-WAX (30 m × 0.25 mm I.D. df = 0.25 µm)
Column Temp.	: 50 ˚C (1.0 min) - 10 ˚C/min - 78 ˚C - 20 ˚C/min - 250 ˚C
Injection Temp.	: 250 ˚C
Carrier Gas	: He
Injection Method	: Split 1:20
Injection Pressure	: 146.2 kPa (3.8 min) - (– 400) kPa/min - 20 kPa (8.28 min)
APC Pressure	: 50 kPa (3.8 min) - 400 kPa/min - 300 kPa (7.97 min)
Injection Volume	: 1.0 µL
Detector	: FID or FTD
Detector Temp.	: 250 ˚C

■ Analysis of Test Solution Spiked with Acrylonitrile Standard

Fig.3 shows chromatograms of the standard addition sample spiked with the equivalent of 80 ppm acrylonitrile. In the first analysis where backflushing was not performed, it took about 55 minutes to heat the column to 250 ˚C to drive off the residual contaminants, for a total analysis time of about 60 minutes.

In the second analysis, after elution of acrylonitrile at 3.8 minutes, backflushing is performed for 4.6 minutes to remove residual solvent and high-boiling substances. This technique allows a typical 60-minute analysis to be shortened to 12.4 minutes. The GC analytical conditions which were used when conducting the backflush are shown in Table 1.

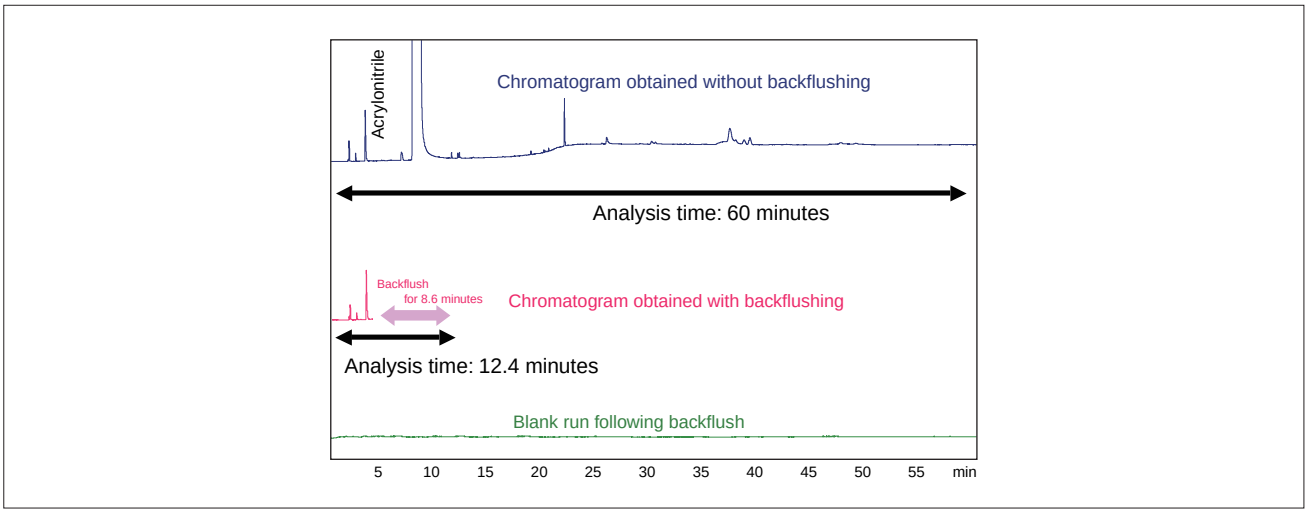

Fig. 3 GC/FID Chromatograms of Sample Spiked with 80 ppm Acrylonitrile

Fig. 4 shows the chromatogram obtained from analysis of an 8 ppm acrylonitrile-spiked sample solution using backflushing with an FTD detector. The FTD is a detector which displays high selectivity and high sensitivity with respect to nitrogen-containing compounds. However, introduction of nitrogen-containing solvents can adversely affect the stability of the detector. By backflushing when N,N-dimethylformamide is used as the solvent, the analysis time is shortened and, at the same time, deterioration of detector stability is prevented.
In addition, the effectiveness of the backflush in removing unwanted contaminants can be verified by the absence of detected peaks in the subsequent blank analysis.

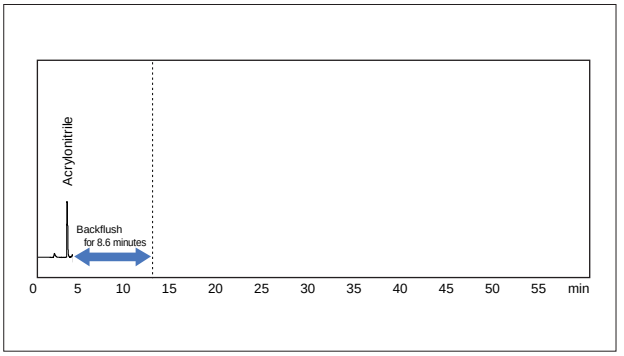

Fig. 4 GC/FTD Chromatogram of Sample Spiked with 8 ppm Acrylonitrile

[References]
Food Sanitation Inspection Guidelines, Physics and Chemistry, 2005, with Ministry of Health, Labour and Welfare Supervision, Japan Food Hygiene Association

NOTES:
✻This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.

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Printed in Japan 3295-12902-10A-IK