

Application News

Gas Chromatography

No. G267

Fast Analysis of VOCs in Polystyrene Using a Capillary Backflush GC System

Residual organic solvents in food packaging materials is a problem that receives serious attention because of the implications to food safety. GC Application News No. G264 addressed this issue, presenting an example of fast analysis of acrylonitrile in ABS

polymer using a backflush GC system.

Polystyrene (PS) is widely used in food packaging materials. In this Application News, we introduce an example of fast analysis of residual volatile organic solvents in polystyrene using a backflush GC system.

■ Overview of VOCs Analysis in PS Polymer

Polystyrene (PS) polymer used in food packaging and cooking utensils contains unreacted monomers and raw material impurities. Due to concern over public health safety and assurance, the substances used in food packaging and cooking utensils are subject to control according to "Section 3: Implements, Containers, and Packaging" of "Specifications and Standards for Food, Food Additives, etc." under Japan's "Food Sanitation Act". Among the regulated substances, five are volatile organics: styrene, toluene,

ethylbenzene, isopropylbenzene, and n-propylbenzene. In the test for volatile substances in polystyrene, the total content of styrene, toluene, ethylbenzene, isopropylbenzene, and n-propylbenzene must not exceed 5 mg/g, however, in the case of polystyrene used in hot water applications, the total content of these volatile substances must not exceed 2 mg/g. Further, the concentrations of styrene and ethylbenzene individually must not exceed 1 mg/g.

■ Backflush GC System

In a backflush GC system, a special backflush element, consisting of an advanced pressure controller (APC) and a connection fitting, are connected at the outlet of the GC column. 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 injector is simultaneously lowered. These pressure changes reverse 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 port split vent. This technique shortens the analysis time, protects the column, and prevents contamination of the detector.

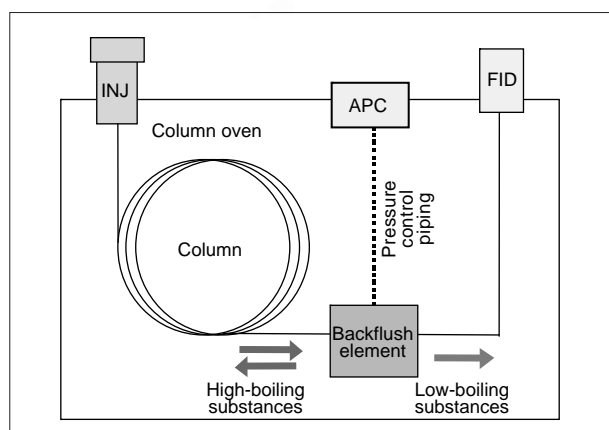


Fig. 1 Backflush GC System

■ Analysis Method (Polystyrene Polymer Pretreatment)

Pretreatment was conducted according to that specified in "Section 3: Implements, Containers, and Packaging" of "Specifications and Standards for Food, Food Additives, etc." under Japan's "Food Sanitation Act".

The sample solution was prepared by first weighing out 0.5 g of shavings from a commercial polystyrene polymer spoon into a measuring flask, and adding enough tetrahydrofuran (THF) to dissolve the

shavings. Then, 1 mL of an internal standard solution consisting of 1 μ L/mL of diethyl benzene (in THF solvent) was added to the flask, and the total volume was brought to 20 mL by adding THF.

Analysis was conducted by GC/FID using a Restek Rtx-Wax column (internal diameter 0.25 mm, length 30 m, film thickness 0.5 μ m).

The pretreatment method is shown in the flow chart of Fig. 2.

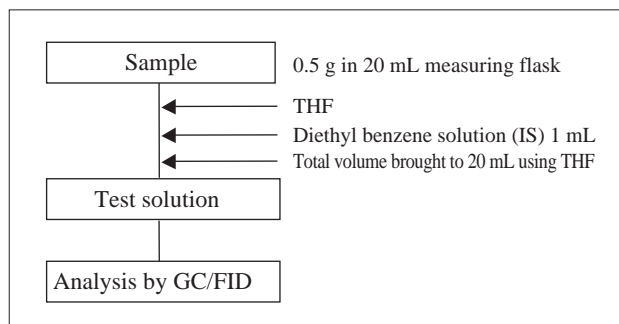


Fig. 2 Preparation of PS Polymer Sample

■ Analysis of Standard Solution

The standard solution of VOCs was prepared by adding diethyl benzene internal standard solution (1 $\mu\text{L}/\text{mL}$). Fig. 3 shows the chromatogram obtained from measurement of the standard solution containing 25 $\mu\text{g}/\text{mL}$ of each substance.

Column	: Rtx-Wax (30 m \times 0.25 mm I.D. df = 0.5 μm)
Column Temp.	: 60 $^{\circ}\text{C}$ - 4 $^{\circ}\text{C}/\text{min}$ - 100 $^{\circ}\text{C}$ - 10 $^{\circ}\text{C}/\text{min}$ - 120 $^{\circ}\text{C}$ - 20 $^{\circ}\text{C}/\text{min}$ - 250 $^{\circ}\text{C}$
Injection Temp.	: 220 $^{\circ}\text{C}$
Carrier Gas	: He
Injection Method	: Split 1: 30
Injection Pressure	: 177 kPa (12 min) - 400 kPa/min - 20 kPa (7.61 min) 50 kPa (12 min) - 400 kPa/min - 300 kPa (7.37 min)
Injection Volume	: 1.0 μL
Detector	: FID
Detector Temp.	: 280 $^{\circ}\text{C}$

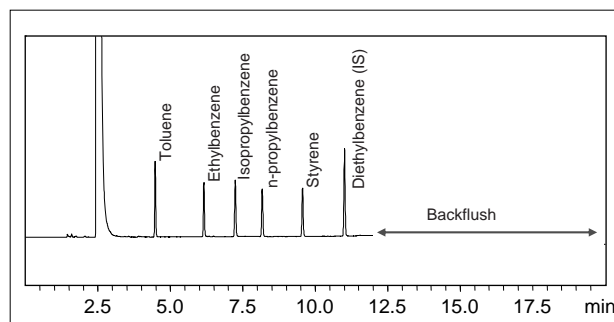


Fig. 3 Chromatogram of Standard Solution

■ Analysis of Sample Solution

Chromatograms obtained from measurement of the prepared sample solution of the commercial polystyrene spoon are shown in Fig. 4; the analytical conditions are shown in Table 1.

When backflush is not conducted, it takes about 60 minutes for the impurities to be driven off with the column temperature at 250 $^{\circ}\text{C}$, for a total analysis time of 80 minutes.

By starting the backflush operation following elution of the target components at 12 minutes from the start of analysis, the 80-minute analysis time was reduced to just 20 minutes by removing the high-boiling impurities, even including the time for elution of the other unnecessary impurities. Further, the fact that no peaks are detected in the blank run conducted after the backflush demonstrates complete removal of the unnecessary substances due to the backflush operation.

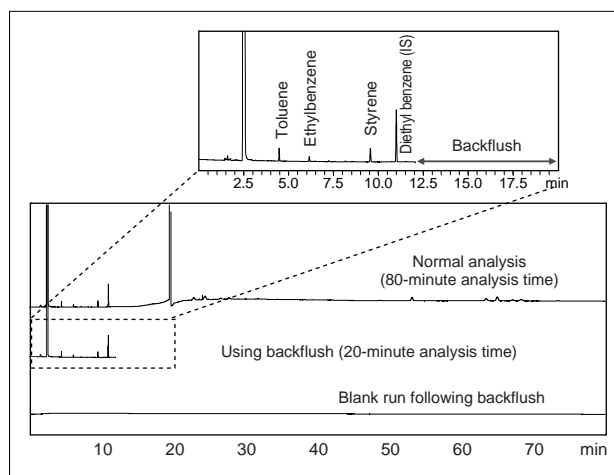


Fig. 4 Chromatograms of Sample Solution

[References]

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

Food Sanitation Act - Specifications and Standards for Food, Food Additives, etc., Section 3: Implements, Containers, and Packaging