

# Application News

Gas Chromatography Mass Spectrometry

No.M251

## Analysis of Food Preservatives by GC/MS

### Introduction

Food preservatives are used to prevent adverse changes and rotting of foods by suppressing the proliferation of microorganisms; they are used in accordance with the properties of the food to which they will be added. Many of the analytical methods used for analyzing food preservatives employ extraction of the compounds of interest by the steam distillation technique, followed by quantitative analysis using high performance liquid chromatography (HPLC). Gas chromatography can also be used for measurement of many compounds used as food preservatives, including benzoic acid, sorbic acid, methyl p-hydroxybenzoate (PHBA methyl), ethyl p-hydroxybenzoate (PHBA ethyl), propyl p-hydroxybenzoate (PHBA propyl), and butyl p-hydroxybenzoate (PHBA butyl), and therefore, gas chromatograph / mass spectrometer (GC/MS) is an effective analysis technique for verifying the detection results obtained by HPLC. This Application News introduces an example of GC/MS analysis of six types of food preservatives.

Table 1 Analytical Conditions

Model	: GCMS-QP2010 Plus
-GC-	
Column	: Rtx-5MS (30 m × 0.25 mm I.D. df = 0.25 μm)
Column Temp.	: 60 °C - 15 °C/min - 280 °C (20 min)
Carrier Gas	: He, 45.0 cm/sec
Carrier Gas Mode	: Constant Linear Velocity Mode
Inj Temp.	: 250 °C
Injection Method	: Split Injection
Split Ratio	: 10:1
Injection Volume	: 1 μL
-MS-	
I.F. Temp.	: 260 °C
I.S. Temp.	: 230 °C
Ionization	: EI
Scan Range	: <i>m/z</i> 40-300
Scan Interval	: 0.3 sec

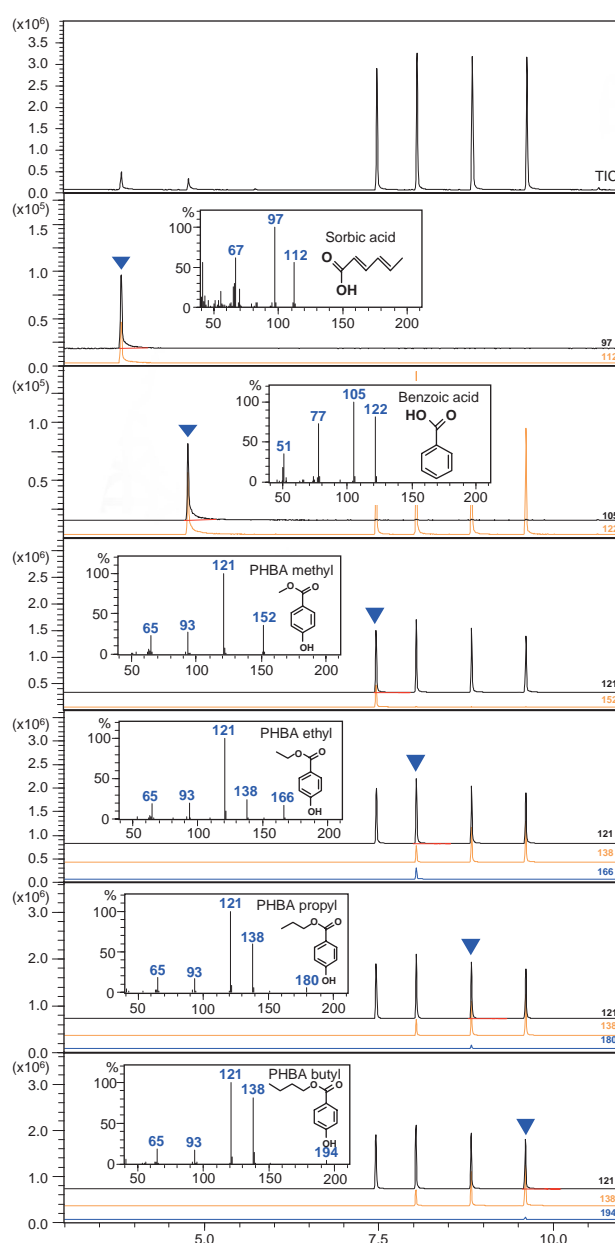


Fig. 1 Chromatograms of 6 Preservatives

## Results

### Standard Samples

Each of six preservatives was used to prepare a 10 mg/L standard solution, and these were analyzed using the analytical conditions shown in Table 1. The resulting chromatograms are shown in Fig. 1. The uppermost data is the TIC chromatogram, and the remaining chromatograms are the mass chromatograms corresponding to the characteristic  $m/z$  of each of the substances; the mass spectrum of each substance is also shown on the corresponding mass chromatogram. Although sensitivities for benzoic acid and sorbic acid were lower than those for the PHBA esters, the peaks for benzoic acid and sorbic acid were clearly detected.

### Soft Drinks

Many commercially available soft drinks contain sodium benzoate as a preservative, and one such soft drink in which sodium benzoate was indicated as a preservative ingredient was analyzed as part of this study. The pretreatment consisted only of diluting the sample 50 to 1 with ethanol; the diluted sample was analyzed directly. The analytical results indicate the detection of benzoic acid in the mass chromatogram (Fig. 2).

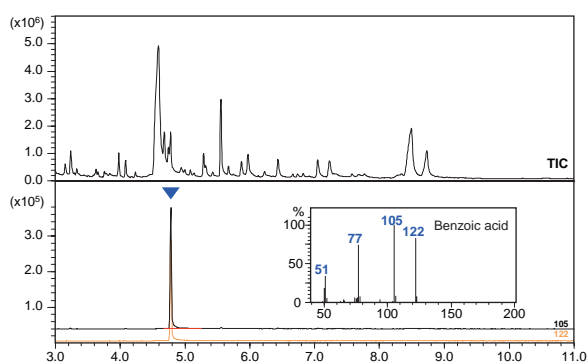


Fig. 2 Result of Soft Drink Analysis

### Liquid Condiment

In Japan, the use of PHBA esters is permitted in liquid condiments such as soy sauce. A liquid condiment sample spiked with 5  $\mu\text{g/g}$  of PHBA esters was also analyzed as part of this study. For the pretreatment, the sample was diluted 10 to 1 with ethanol and centrifuged for 5 minutes at 1000 rpm. The precipitate was removed, and the supernatant solution was analyzed without any further pretreatment. The analytical results are shown in Fig. 3. The mass chromatograms of the added PHBA esters demonstrate that they were clearly detected.

Many chromatographic peaks were observed in the TIC chromatograms of both the soft drink and the liquid condiment. Although the compounds of interest are difficult to observe in the TIC, the target substances are clearly detected in the corresponding mass chromatograms. Furthermore, mass spectra of the preservatives were easily obtained using spectrum subtraction. These results demonstrate that the preservative could be detected not only in the standard sample but in the actual samples as well using a simple pretreatment procedure. Clearly the results of this study show that analysis by GC/MS is effective as a confirmation technique for these substances.

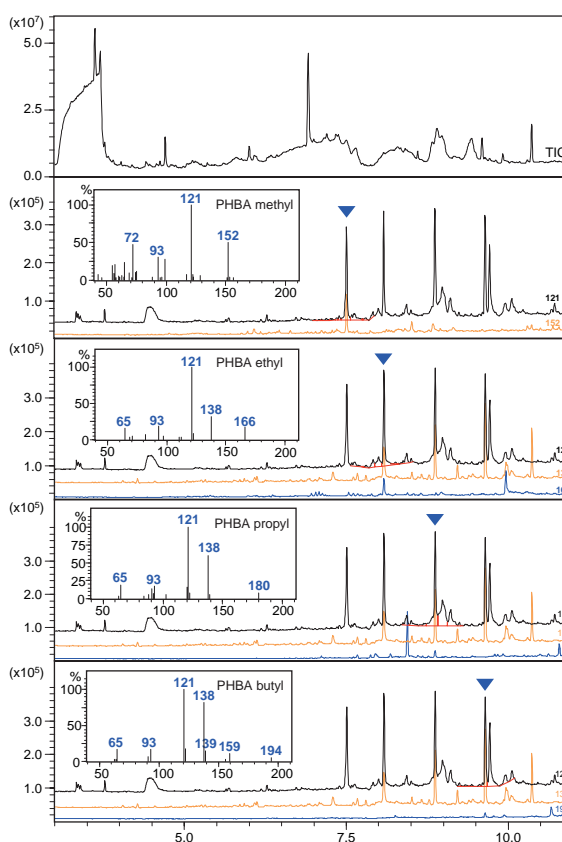


Fig. 3 Chromatograms of Soy Sauce Spiked with 4 kind of Preservatives

#### 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.



SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax: 81(3)3219-5710  
Cable Add.: SHIMADZU TOKYO