SHIMADZU APPLICATION NEWS

LAAN-A-G-E009

● GAS CHROMATOGRAPHY

No.G245

Analysis of Thiophene in Benzene by GC-FPD

Analysis of thiophene in benzene has long been conducted as part of the purity control for benzene. Recently, with the demand for higher purity benzene, there is increased demand for low concentration analysis for thiophene.

Thiophene is a sulfur-containing five-membered heterocyclic compound, which is typically analyzed using an FPD operated in the sulfur mode (S-mode). Since the boiling point of thiophene is close to that of benzene, insufficient chromatographic separation of benzene and thiophene often occurs especially when analysis times are short, and then an FID is not available. Therefore, analysis is conducted using the selectivity of the FPD, carrying over the incomplete separation. However, high-sensitivity analysis of low concentration thiophene in benzene may not be possible, even using the FPD, because of the effect of quenching due to the principle constituent, benzene. The reduction of quenching by changing the FPD flow rates of hydrogen and air and the use of a thick-film capillary column to achieve high sensitivity analysis is the subject of this investigation.

■ Change in Benzene Response with Changes in Detector Gas Flow Rates

First, the change in intensity of the benzene and thiophene peaks as a result of variable detector gas flow rates (hydrogen, air) was investigated using the FPD-2014 with a packed column. As shown in Fig.1, multiple negative benzene peaks are generated which overlap with thiophene. These benzene interferences make high-sensitivity analysis of thiophene impossible under recommended conditions using the FPD-2014 (S mode) with recommended detector gas flow rates (hydrogen 90 mL/min, air 80 mL/min) and a packed column. When the flow rates are changed to 90 mL/min and 120 mL/min for hydrogen and air, respectively, the sensitivity is slightly diminished, but thiophene is confirmed at 0.1 ppm.

Increasing the liquid phase percentage is normally required in order to achieve adequate separation of benzene and thiophene. However, this approach is unsuitable for high sensitivity analysis due to peak broadening resulting from diffusion of the analyte within the liquid phase when the liquid phase percentage is increased. Moreover, even increasing the injection volume to 5 µL or greater in order to increase sensitivity is ineffective because the peak width of benzene becomes wider, adversely affecting separation from thiophene.

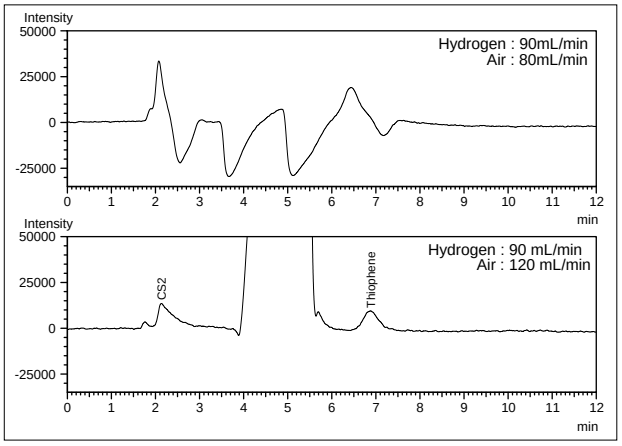

Fig.1 Analysis with a Packed Column

Table 1 Analytical Conditions

Model	: GC-2014
Column	: 5 % Thermon 3000 ChromosorbW AW-DMCS 80/100 mesh 3 m × 3.2 mm I.D. Glass
Column Temp.	: 50 ˚C
Carrier Gas	: He 25 mL/min
Inj. Temp.	: 200 ˚C
Detector	: 230 ˚C FPD-2014(Packed nozzle)
Injection volume	: 5 µL
Sample	: 0.1 ppm Thiophene / Benzene

■ Analysis using GC-2014 with a Wide Bore Column

The use of a wide-bore capillary column was then investigated. The packed column injector was used with a WBC attachment for connection to the wide bore column. By using a wide- bore capillary column at least 25 m in length and a minimum film thickness of 3 µm (corresponding to PEG-20M), the extent of benzene quenching can be reduced by changing the hydrogen and air flow rates to enable thiophene analysis at 0.1 ppm. The high sensitivity analysis of thiophene with better chromatographic peak shape than with the packed column was achieved. The quartz packed column nozzle can be replaced with a SUS capillary column nozzle in the FPD-2014. Fig.2 and Fig.3 show analysis examples using the packed and capillary nozzles, respectively. Some change in the hydrogen and air flow rates relative to those utilized in the analysis example may be required due to the differences in column insertion length and shield ring height. In the example, analysis was conducted using the standard positions of column insertion and shield ring. For fine adjustment of detector gas flow rates, the addition of an optional electronic flow controller (Optional APC for FPD-2014, P/N: 221-47742-81) is recommended.

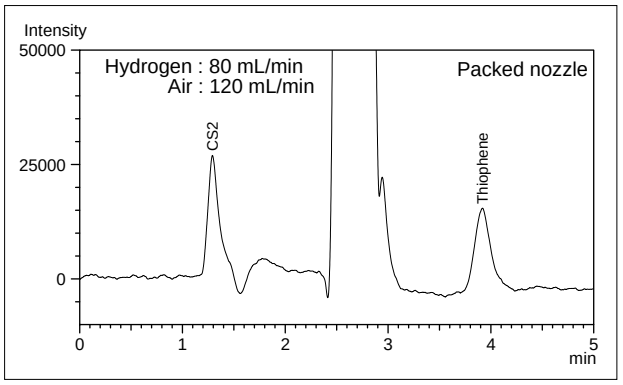

Fig.2 Analysis by FPD-2014 with Packed Nozzle

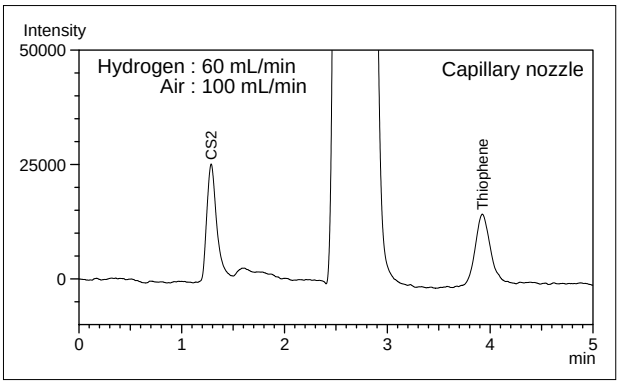

Fig.3 Analysis by FPD-2014 with Capillary Nozzle

Table 2 Analytical Conditions (Fig.2 and Fig.3)

Model	: GC-2014
Column	: 007-CW 25 m × 0.53 mm I.D. d.f.=3.0 µm
Column Temp.	: 75 ˚C
Carrier Gas	: He 7 mL/min
Inj. Temp.	: 200 ˚C
Detector	: 230 ˚C FPD-2014+APC
Injection volume	: 3 µL
Sample	: 0.1 ppm Thiophene / Benzene

■ Analysis using GC-2010 with a Wide Bore Column

Analysis was also conducted using a GC-2010 with the wide bore capillary column described previously. A slight difference in the detector gas flow rates is due to the difference in the FPD-2010 and FPD-2014 nozzle shapes. Although a wide bore capillary column was used, analysis was possible with the FPD-2010 with the shield ring of standard position. Flame extinction did not occur even with a 5 µL injection volume. Moreover, high sensitivity analysis of 0.1 ppm of thiophene was achieved with a 1 µL injection.

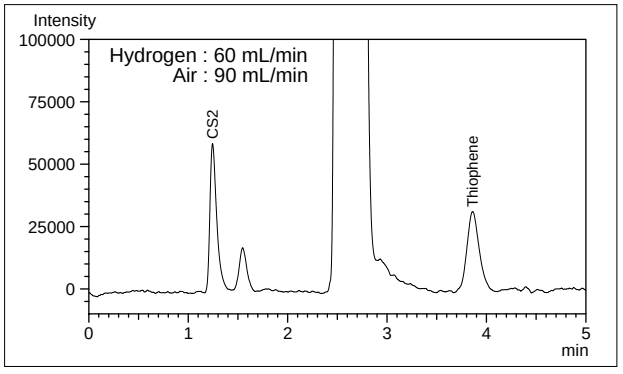

Fig.4 Analysis using FPD-2010

Table 3 Analytical Conditions (Fig.4)

Model	: GC-2010
Column	: 007-CW 25 m × 0.53 mm I.D. d.f.=3.0 µm
Column Temp.	: 75 ˚C
Carrier Gas	: He 7 mL/min
Inj. Temp.	: 200 ˚C
Detector	: 230 ˚C FPD-2010
Injection volume	: 1 µL
Sample	: 0.1 ppm Thiophene / Benzene

■ Recommended Columns

Quadrex Corp. 007-CW 25 m × 0.53 mm I.D. df=3.0 µm (P/N : 335-625 Shimadzu GLC)
30 m × 0.53 mm I.D. df=3.0 µm (P/N : 335-627 Shimadzu GLC)
BTR-CW 25 m × 0.53 mm I.D. df=3.0 µm (P/N : 335-814 Shimadzu GLC)
30 m × 0.53 mm I.D. df=3.0 µm (P/N : 335-817 Shimadzu GLC)

Although analysis was possible using columns with film thicknesses of 4 µm and 5 µm, analysis times became longer.

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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