SHIMADZU

Application News

Gas Chromatography No.G266A

Detector-Switching Analysis Using a Capillary Switching Device

A capillary switching device allows switching of flow lines with high accuracy. This device can be used to switch between 2 detectors, allowing concurrent analyses with multiple detectors to be conducted easily. Detector-switching analysis is different from detector-splitting analysis, so when analysis is conducted using detector switching, the entire sample is flowing into the appropriate detector, and accurate information can be obtained from multiple detectors during a single analysis run without sacrificing sensitivity. (With the detector splitting technique, only a specific fraction of the sample is directed to each of the detectors during the entire analysis.)

A switching program can easily be created with special software that can be downloaded from the Shimadzu website free of charge.

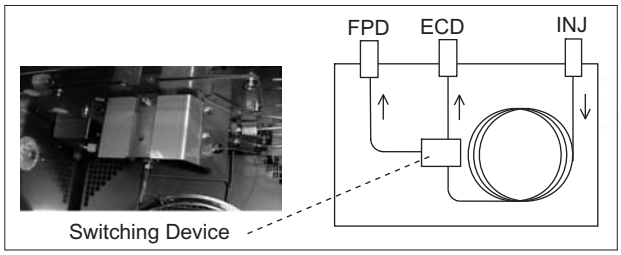

Fig. 1 Device Configuration for Detector-Switching Analysis

■ Analysis of Pesticides Using ECD - FPD Switching

When conducting residual pesticides analysis by GC, a detector with high sensitivity and good selectivity is typically used. Although such a detector is highly effective for analysis of certain contaminants in agricultural products, analysis using multiple detectors is required for detection of all the pesticide constituents. Here we introduce an example of simultaneous analysis of a standard solution of pesticides using switching between an FPD (flame photometric detector) and ECD (electron capture detector).

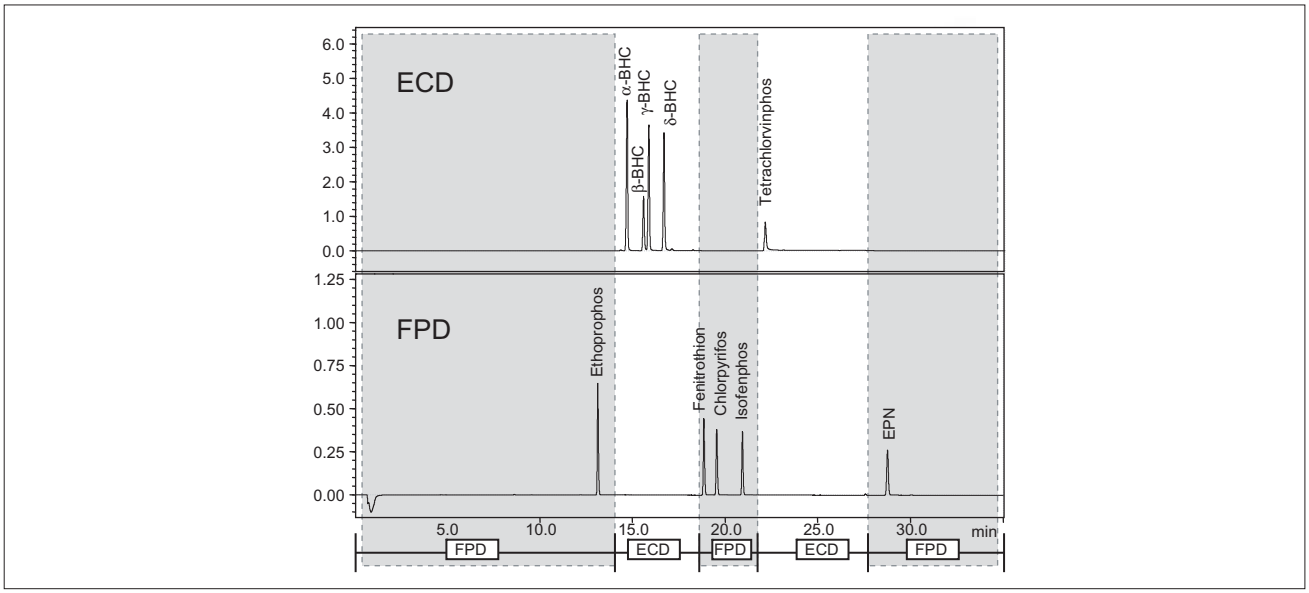

Fig. 2 Chromatograms of Pesticides Obtained by ECD - FPD Switching

Table 1 Analytical Conditions

Instrument	: GC-2010 Plus	Injection Method	: Splitless
Column	: Rtx-5MS (30 m × 0.25 mmI.D. df = 0.25 µm)	Sampling Time	: 1 min (High Pressure: 350 kPa, 1 min)
Column Temp.	: 80 °C (1 min) - 20 °C/min - 180 °C - 5 °C/min - 280 °C (9 min)	Detector	: ECD: 300 °C (1nA), Make-up: N_2 60 mL/min, FPD: 300 °C, H_2: 80 mL/min, Air: 120 mL/min
Carrier Gas	: He (150 kPa, Constant Pressure)		
Switching Press.	: 90 kPa	1st Restrictor (ECD side)	: 0.5 m × 0.18 mmI.D.
Injection Port	: 250 °C	2nd Restrictor (FPD side)	: 0.5 m × 0.15 mmI.D.
Sample	: 0.1 mg/L, 2 µL injection		

■ Solvent - Elimination Analysis

Due to the problem of differing sensitivity among selective detectors, it is advisable to prevent some solvents or substances from flowing into a detector, and sometimes these substances cannot be eliminated from the sample. By using a switching device, unnecessary constituents can be discharged to waste without allowing them to be introduced into the detector. Here we introduce an example of analysis in which a solvent (dichloromethane) is eliminated before entering the detectors; dichloromethane accelerates deterioration of the FTD alkaline source and also has a high response by ECD.

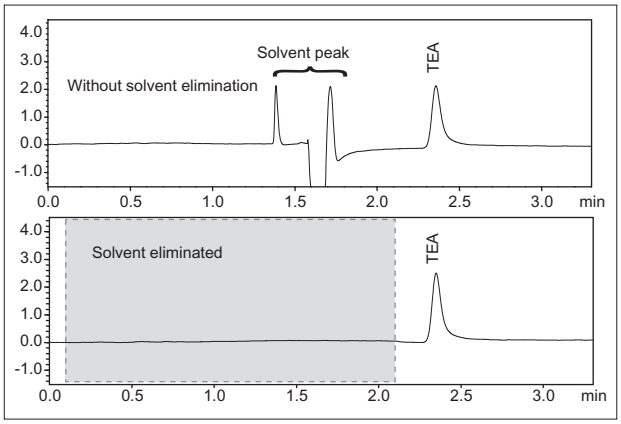

Fig. 3 Chromatograms of With and Without Solvent Elimination

Table 2 Analytical Conditions

Instrument	: GC-2010 Plus	Injection Method	: Split, Split Ratio: 1:15
Column	: Rtx-1 (30 m × 0.32 mmI.D. df = 5 µm)	Detector	: FTD: 260 °C 1 pA, H_2: 1.5 mL/min, Air: 145 mL/min, Make-up Gas: He27.5 mL/min
Column Temp.	: 150 °C		
Carrier Gas	: He (204.2kPa, Constant Pressure)	1st Restrictor (ECD side)	: 0.5 m × 0.18 mmI.D.
Switching Press.	: 90 kPa	2nd Restrictor (Vent side)	: 0.5 m × 0.15 mmI.D.
Injection Port	: 250 °C		

■ Air Elimination Analysis

The headspace-ECD (HS-ECD) method is used for analysis of VOCs in water. In this method, both the air that is in vial as well as the volatile components are injected. However, since the sensitivity tends to fluctuate when oxygen is introduced into the ECD, this can adversely affect detection stability over time. Using a switching device to discharge air prior to reaching the detector can extend the stability of the detector used in the HS-ECD method.

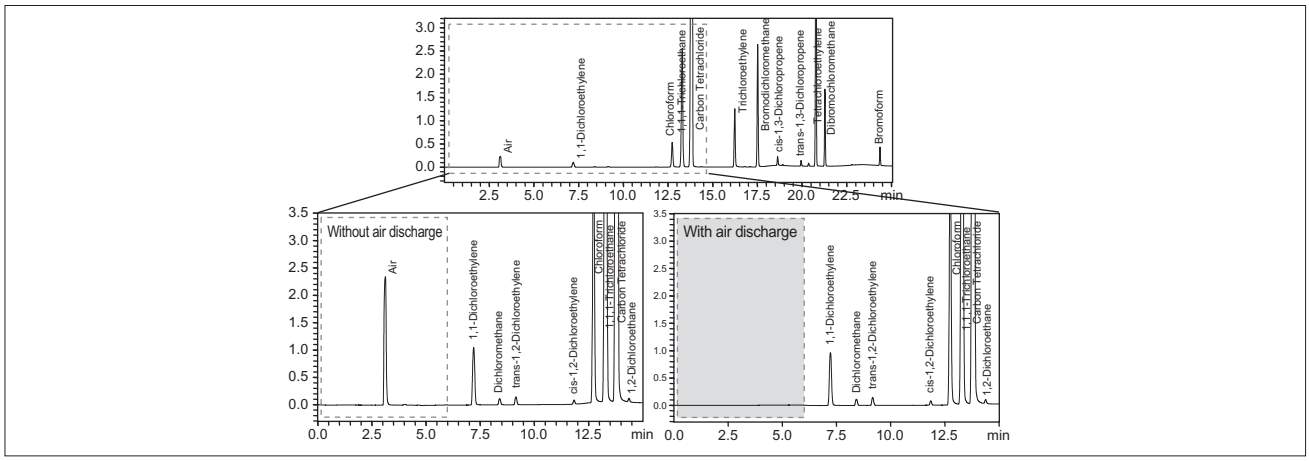

Fig. 4 Chromatograms With and Without Air-Elimination

Table 3 Analytical Conditions

Instrument	: GC-2010 Plus + TurboMatrix HS40	HSVial	: 60 °C (60 min) Injection Time : 0.1 min HS Press : 250kPa
Column	: DB-624 (60 m × 0.32 mmI.D. df = 1.8 µm)	Injection Method	: Split Split Ratio : 1:4
Column Temp.	: 40 °C (5 min) - 4 °C/min - 80 °C - 10 °C/min - 220 °C (3 min)	Detector	: ECD 250℃1nA Make-up Gas : (N_2) 60mL/min
Carrier Gas	: He (234.4 kPa, Constant Pressure)	1st Restrictor (ECD side)	: 0.5m × 0.18mmI.D.
Switching Press.	: 90 kPa	2nd Restrictor (Vent side)	: 0.5 m × 0.15 mmI.D.
Injection Port	: 200 °C		

SHIMADZU

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