SHIMADZU APPLICATION NEWS

LAAN-A-G-E002

● GAS CHROMATOGRAPHY

No.G231

Optimum Initial Column Temperature in Splitless Injection Analysis Using Various Solvents (Part 2)

Use of Open Tubular Capillary Column (Guard Column)

In Application News G230, we discussed the optimum initial column temperature in splitless injection analysis using various solvents, using an analytical column of 0.25mm I.D. × 30m long and df = 0.25µm.
In pesticide analysis, a deactivated open tubular capillary column (guard column) is sometimes connected before the analytical column to help protect the analytical column from contamination. This Application News investigates the differences in peak shape and separation obtained at different initial column temperatures in splitless analysis using the solvents acetone, hexane, acetonitrile and toluene when a guard column is connected to the analytical column.
We connected a deactivated 0.53mm × 50cm guard column before the analytical column using a press-tight connector. An organophosphorus standard mixture source solution (10mg/L each acetone solution) was diluted by a factor of 100 with each of acetone, hexane, acetonitrile and toluene, and the resultant solutions were used as the sample solutions (each constituent 0.1mg/L). The sample solutions were subjected to splitless analysis at different initial column temperatures to evaluate the peak shapes etc. The other conditions were same for all the samples.

Fig.1 and 2 show the chromatograms of the acetone and hexane solutions, respectively, obtained with initial column temperatures from 35˚C to 100˚C. Fig.3 and 4 show the chromatograms of the acetonitrile and toluene solutions, respectively, obtained with initial column temperatures from 50˚C to 120˚C. The peak shape quality in these chromatograms was evaluated, and the results are summarized in Table 1.

When the guard column was not connected, peak splitting and or widening occurred with an initial column temperature of 35˚C for the acetone solution, 50˚C to 100˚C for the acetonitrile and toluene solutions (Application News G230). However, when the guard column is connected, no peak splitting or widening is observed, although some peak tailing is observed with lower initial column temperatures.

In splitless analysis, after sample injection, solvent re-condensation occurs easily at the front tip of the column. If there is too much of this condensed liquid, some of it will move through the column in liquid form, while sending the solute downstream (flooding phenomenon), causing abnormal peak shapes.[1)] If a guard column without a liquid phase is connected before the analytical column, even if a portion of the condensed solvent at the tip of the column moves through the column with the solute, the solute does not move exceeding the length of the guard column. Because there is no liquid phase in the guard column, even if the target component is present broadly, it vaporizes and moves quickly when being heated, causing no peak splitting.
At higher initial column temperatures, peak tailing was reduced for DDVP and dimethoate (60˚C and above for acetone and hexane solutions, and 80˚C and above for acetonitrile and toluene solutions). When the guard column was connected, better peak shapes were obtained at higher initial column temperatures.

The effect on preventing peak tailing and splitting depends not only on the inner diameter, length and manufacturer of the deactivated guard column, but also the inner diameter, length and film thickness of the analytical column, as well as sample injection volume and column inlet pressure. Thus all the results obtained here do not apply to all splitless analyses.

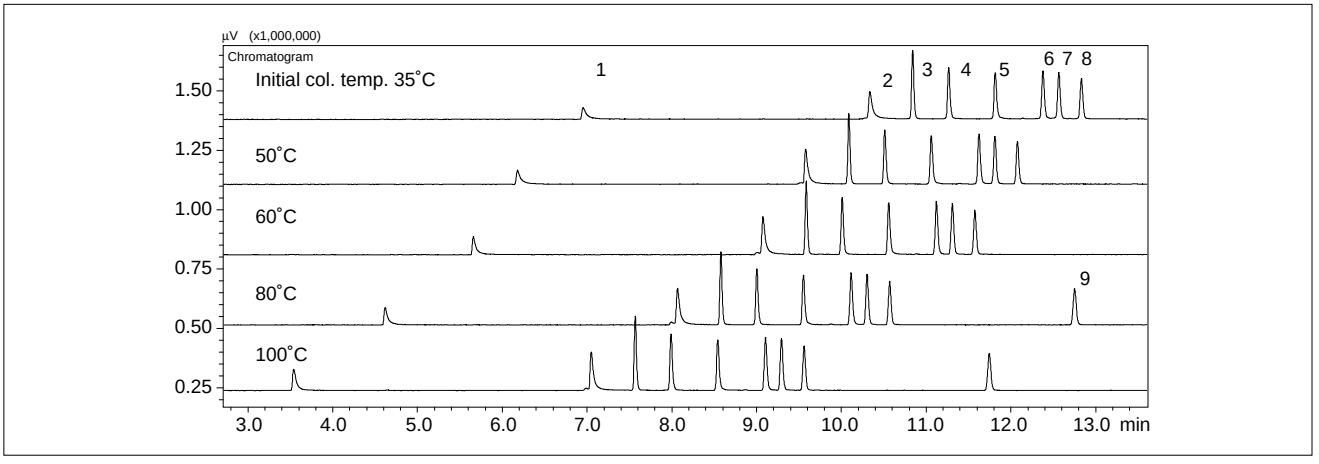

Fig.1 Chromatograms of Organophosphorus Pesticides in Acetone Solution (0.1mg/L each)
(Peak Name 1:DDVP, 2:Dimethoate, 3:Diazinon, 4:IBP, 5:Parathion-methyl,6:MEP, 7:Malathion, 8:Chlorpyrifos, 9:Prothiofos)

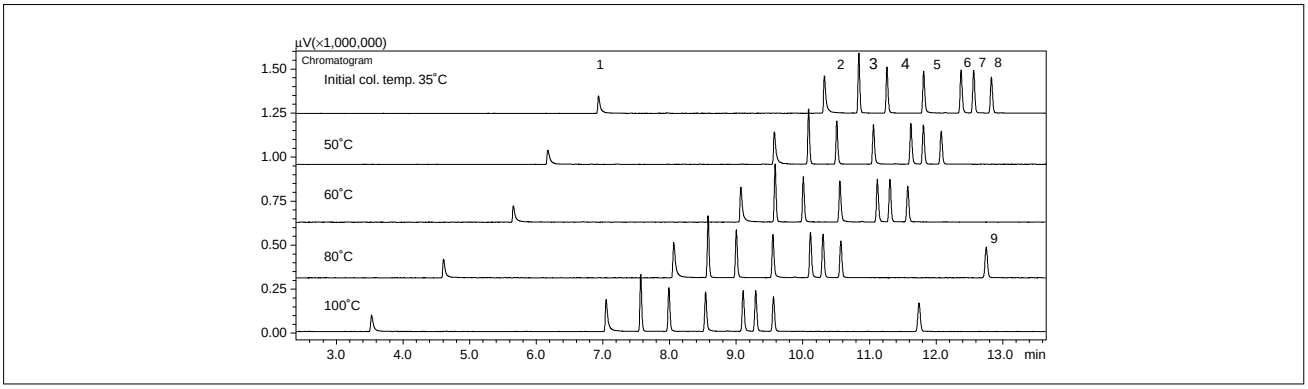

Fig.2 Chromatograms of Organophosphorus Pesticides in Hexane Solution (0.1mg/L each) (Peak name : see Fig.1)

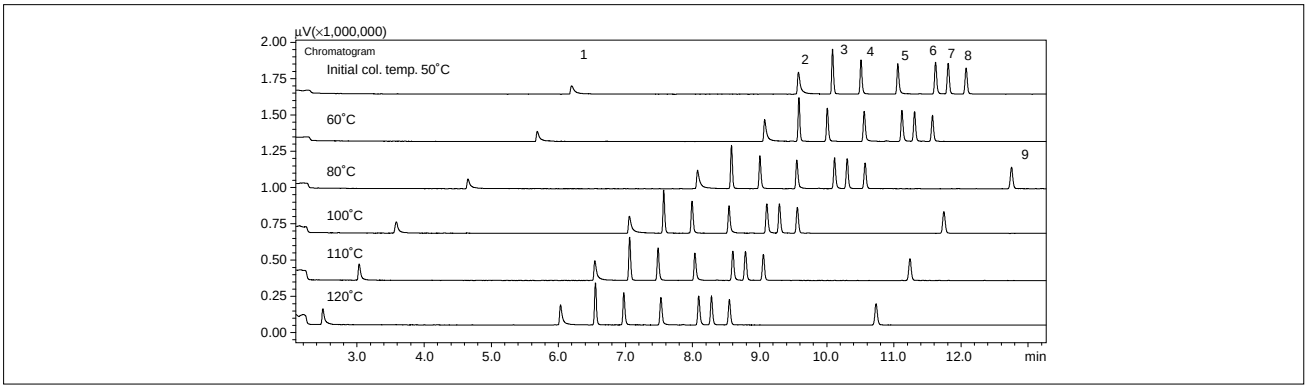

Fig.3 Chromatograms of Organophosphorus Pesticides in Acetonitrile Solution (0.1mg/L each) (Peak name : see Fig.1)

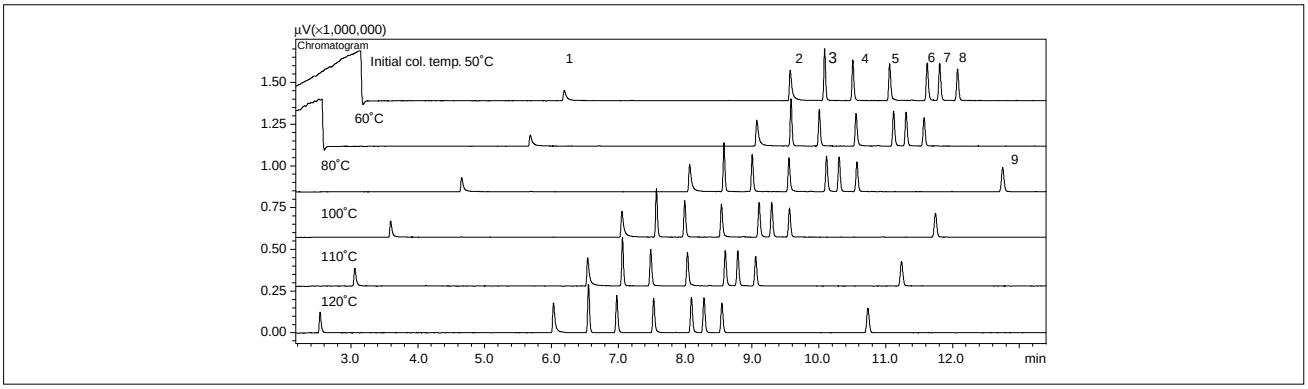

Fig.4 Chromatograms of Organophosphorus Pesticides in Toluene Solution (0.1mg/L each) (Peak name : see Fig.1)

Table 1 Relationship between Initial Column Temperature and Peak Shape in Splitless Analysis Using Different Solvents

	Boiling point	Initial Column Temperature							Initial Temp. for Good Peak Shape
		35˚C	50˚C	60˚C	80˚C	100˚C	110˚C	120˚C	
Acetone	56.5˚C	●	O	O	O	O	–	–	50~100˚C
Hexane	69˚C	O	O	O	O	O	–	–	35~100˚C
Acetonitrile	81.6˚C	–	●	●	●	O	O	O	100~120˚C
Toluene	110.6˚C	–	●	●	●	O	O	O	100~120˚C

O: Good
–: Not evaluated
●:Initial column temperature where peak shape was poor using analytical column only

Analytical Conditions

Model	: GC-2010AFFp AOC-20i
Column	: Deactivated open tube column 50cm × 0.53mm I.D. (Manufactured by Restek)* + Rtx-5ms 30m × 0.25mmI.D. df=0.25μm
Column Temp.	: 35~120˚C (1min) -20˚C/min-190˚C (0min) -5˚C/min-260˚C (3min)
Carrier Gas	: He (2.3mL/min) Linear Velocity 45cm/s (Constant Linear Velocity Mode)
DET	: FPD2010 (P-Mode)
Inj.Temp.	: 250˚C
Det.Temp.	: 290˚C
Injection Method	: High Pressure Splitless (350kPa,1min)
Injection Volume	: 1μL
*Deactivated open tube column	: 0.53mmI.D. × 5m P/N10045 (Manufactured by Restek, sold by Shimadzu GLC)
Press-tight connector	: P/N221-38102-91

References: 1) Sample Introduction Technique Guidebook in CGC, Translated by Gas Chromatography Research Discussion Group, Maruzen Publishing (1999)

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