Screening for Pesticides with Two Columns in a Split/Splitless Capillary Injector: A Comparison of Two Techniques for Installing the Columns

Application Note

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Introduction

Since the introduction of split/splitless capillary injectors, chromatographers have been installing two columns in one injector for simultaneous analysis and confirmation. The technique is used in several different ways — sometimes the columns are the same and responses are studied on two different detectors, often the detectors are the same but columns of different polarity are used to obtain different retention times for the components of the sample. Different columns and detectors are also used to obtain the most information. While it may not be necessary or desirable that there be a 50:50 split, it is clearly essential that a repeatable splitting of the sample between the two columns occurs.

Originally, both columns were installed in the injector body, using a two-hole ferrule or a graphite ferrule with a large hole. Light-weight QuickSeal connectors are much easier to use than the low-dead volume connectors that preceeded them and are available as two-way and "Y" connectors to serve as inlet or effluent splitters. In this note, a comparison was made in splitless injection of two methods of installing two columns — inserting both through a single ferrule and using a "Y" connector.

A test sample containing halogenated and organophosphorus pesticides and nitrogen herbicides of various volatilities was prepared. Particular attention was given to the organophosphorus pesticide, Zolone. This compound was selected because it is relatively non-volatile and elutes late in the run. It tends to show discrimination and poor repeatability when determined by hot splitless injection.

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Instrumentation

A GC was used, equipped with nitrogen-phosphorus and electron capture detectors (TSD and ECD), a 1177 Split/Splitless Injector, Agilent Galaxie chromatography software for data handling, and autosampler. The autosampler held 1 µL samples with upper and lower air gaps and 1 µL solvent plug (hexane) injected at 0.5 µL/s, with needle residence time of 2 s.

Conditions

Columns:	VF-17ms (connected to the TSD), 15 m x 0.25 mm, df=0.25 µm (part number CP8979) VF-5ms (connected to the ECD), 15 m x 0.25 mm, df=0.25 µm (part number CP8939)
Column Temp:	40 °C, 20 °C/min to 280 °C, hold
Injector Temp:	280 °C, open splitter at one minute
Carrier Gas:	Helium at 59 cm/s, splitter flow 25 mL/min
Detection:	300 °C, TSD, Range 10-12, ECD, Range 10

Sample Preparation

The sample contained several halogenated pesticides at 0.2 ng/µL and nitrogen and phosphorus containing pesticides at 2 ng/µL in hexane.

The two columns were connected to the injector using two different techniques.

- Columns inserted into the injector by passing them both through a 0.8 mm graphite ferrule.
- Columns connected to a QuickSeal "Y" splitter and a 50 cm piece of the VF-17ms column was used to connect the splitter to the injector.

Results and discussion

Table 1 compares area count precision using the two methods of installing two columns for several of the pesticides throughout the run. Note that repeatability of area counts deteriorated for the later eluting compounds, but there was consistent distribution between the two columns (Figure 1). Although the splitting between the two columns occurred further down the path with the "Y" connector, the results seemed to be comparable with the two techniques. Installation of two columns was easier with the "Y" connector but these connectors are quite expensive. In addition, an extra piece of fused silica tubing was required, and it must be ascertained that this material is inert. Table 2 shows retention times for the two columns.

Table 1. Comparison of area count Precision with two columns in splitless and temperature-programmable (SPI) injectors

Pesticide	Retention time (min)	Area counts x 10^{-2} (%RSD, n=8)		
		Splitless		SPI
		Two columns in one injector	QuickSeal splitter	QuickSeal splitter
Aminocarb[a]	7.79	5676 (1.88)	3416 (1.91)	3177 (1.03)
Endrin[b]	10.06	6771 (1.69)	8082 (2.05)	8451 (1.43)
Zolone[a]	12.54	13347 (6.58)	10119 (5.96)	12061 (3.60)
Co-Ral[a]	13.62	9700 (13.0)	5687 (11.6)	12905 (2.24)

[a]TSD, [b]ECD

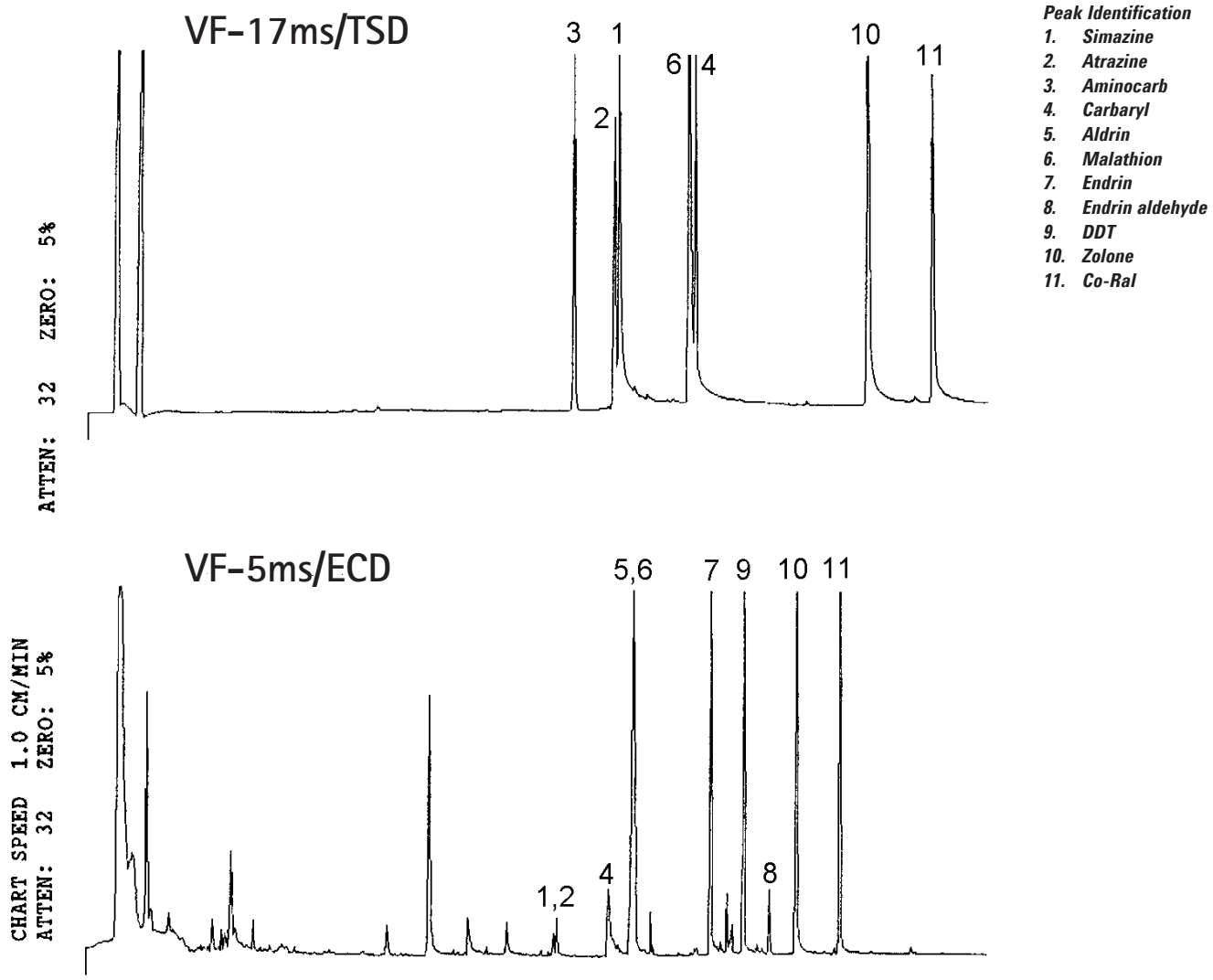

Figure 1. Dual chromatogram of pesticide test sample from the injection into two columns in one splitless injector

Table 2. Calibration data of semi-volatile compounds

Compound	Concentration (ng/µL)	RT (min)	
		VF-5ms/ECD	VF-17ms/TSD
Simazine	2.0	7.56	8.48
Atrazine	2.0	7.65	8.40
Aminocarb	2.0	*	7.77
Carbaryl	2.0	8.44	9.71
Aldrin	0.2	8.77	*
Malathion	2.0	8.77	9.61
Endrin	0.2	10.04	*
Endrin aldehyde	**	10.27	*
DDT	0.2	10.57	*
Zolone	2.0	11.43	12.46
Co-Ral	2.0	12.12	13.50

* No response with this detector, ** Decomposition product

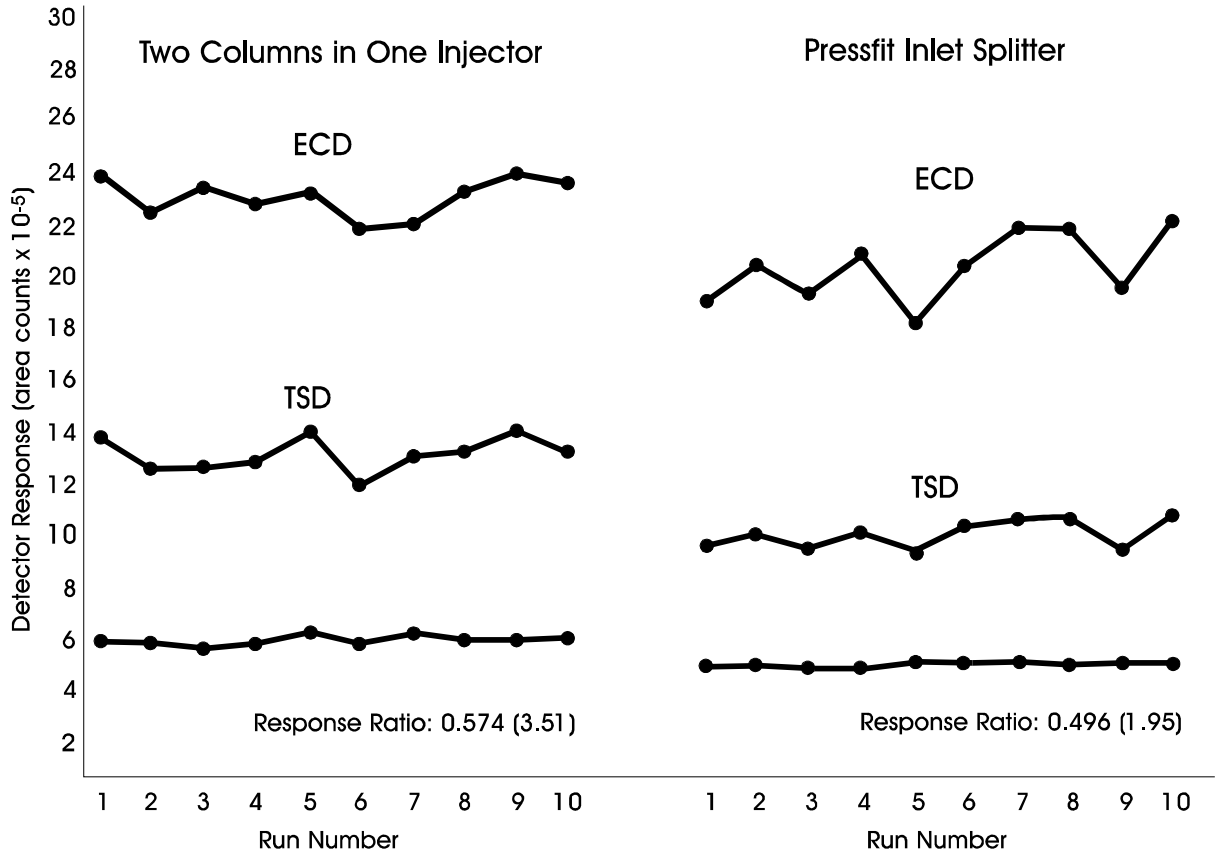

Figure 2. The distribution of Zolone between two 15 m x 0.25 mm columns in splitless injection

Left: Both columns were inserted through one ferrule into the injector
Right: Columns connected to an inlet splitter and one 50 cm length of fused silica inserted into the injector

Each point represents a detector response from a single run. Note that Zolone was a late eluter and particularly subject to mass discrimination, but the precision of the response (bottom of figure in parenthesis) was good.

Conclusion

The QuickSeal connectors work well. For on-column capillary injectors or for certain other capillary injectors such as the Agilent septum-equipped temperature programmable injector they are essential for the two-column technique.

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Published in UK, October 01, 2010
SI-02326

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