

Analysis of Organophosphorus Pesticides by GC

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Key Words

Organophosphorus pesticides, TraceGOLD TG-5MS column, TRACE 1310, US EPA Method 8141B, quartz liner

Abstract

This application note demonstrates the use of a deactivated, splitless quartz liner with single taper and a 5% phenyl polysiloxane phase column for the separation of an organophosphorus pesticides standard mix. This was analyzed on a Thermo Scientific™ TRACE™ 1310 GC equipped with a modular split/splitless (SSL) injector and a flame ionization detector (FID).

Introduction

US EPA 8141B is one of a number of standard analytical methods used for the determination of organophosphorus pesticides (OPPs) in aqueous and solid samples by gas chromatography. OPP can easily degrade in the injector port, which can lead to poor peak profiles. This causes activity within the GC inlet port when repeated injections are made, producing matrix effects. These pesticides can then interact with the active sites and produce peak tailing and poor reproducibility of results.

Using a Thermo Scientific deactivated, packed splitless quartz liner results in a reduction of activity on the surface of the liner, giving excellent reproducibility when compared to several other liner formats. The liner is treated using a proprietary process to reduce any surface activity. These characteristics lead to highly symmetrical peak shapes. In addition, deactivated quartz wool helps in trapping the non-volatile compounds.



This analysis is performed on an ultra-low bleed 5% phenylpolysiloxane phase GC column. The OPP analysis was performed in splitless injection mode using a Thermo Scientific™ TraceGOLD™ TG-5MS 30 m × 0.25 mm × 0.25 µm GC column and a deactivated, packed splitless quartz wool liner for the TRACE 1310 GC, which is equipped with a modular plug and play split/splitless (SSL) injector and a flame ionization detector (FID). This fulfills the requirement of US EPA Method 8141B for the analysis of the OPPs listed in Table 1.

Consumables		Part Number
Column:	TraceGOLD TG-5MS 30 m × 0.25 mm × 0.25 µm	26098-1420
Septum:	BTO coated 11 mm center guide (50/pk)	31303233
Liner:	Splitless liner with single taper 78.5 × 4 × 6.3 mm	453A1925
Column ferrules:	Graphite ferrule for 0.1–0.32 mm i.d. columns 10/pk	290GA139
Injection syringe:	10 µL syringe FN 50 mm T Gauge 26, cone tip	36500525
Vials and closures:	Thermo Scientific 9 mm Wide Opening Screw Thread Vials Convenience Kit, 2 mL Clear Glass Vial with ID Patch, Blue Closure with PTFE/Blue Silicone Septa	60180-599

Solutions

A working standard solution of 20 µg/mL of EPA 8141 was prepared in acetone. The stock solution was obtained commercially at a concentration of 1000 µg/mL.

Separation Conditions		Part Number
Instrumentation:	TRACE 1310 mainframe 230 V GC	14800302
Carrier gas:	Helium	
Split flow:	50 mL/min	
Column flow:	1.2 mL/min, constant flow	
Oven temperature:	40 °C (1 min), 12 °C/min, 280 °C (10 min)	
Injector type:	TRACE 1310 SSL Injector module	29903010
Injector mode:	Splitless	
Injection details:	Splitless (1 min)	
Injector temperature:	220 °C	
Detector details:	TRACE 1310 FID module	29903001
FID parameters:		
Temperature:	280 °C	
Air flow:	350 mL/min	
Hydrogen flow:	35 mL/min	
Nitrogen makeup flow:	30 mL/min	

Injector Conditions

Instrumentation:	Thermo Scientific AS1300 Autosampler
Injection Volume:	1 µL
Wash solvent:	Acetone/hexane (1:1 v/v)

Data Processing

Software:	Thermo Scientific™ Chrom-Card™ data system
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Results

Figure 1 shows the TIC chromatogram for 22 OPPs at 20 µg/µL using a TraceGOLD TG-5MS column and a standard, deactivated, splitless quartz liner for the TRACE 1310 GC instrument. Table 1 shows the peak identification of the OPPs according to their retention times. Table 1 includes the reproducibility data for ten injections. The stationary phase of the TG-5MS GC column, in combination with the deactivated splitless liner, provides excellent performance due to minimal interaction of active compounds with active sites on the column, the glass wall of the liner, or the deactivated quartz wool. This minimizes peak tailing of the OPPs and gives highly symmetrical peak shapes. The combination of a TG-5MS GC column, the deactivated liner, and the TRACE 1310 GC gave excellent injection reproducibility of between 1.7% and 3.4% for the 22 OPPs tested (Table 2).

The tailing factors calculated according to the USP method for all peaks were 0.82–0.97 apart from mevinphos, which gave a tailing factor of 0.77. The resolution value between peaks 17 and 18 was 1.75 according to the USP criteria. For peaks 12 and 13, the calculated resolution was 0.90.

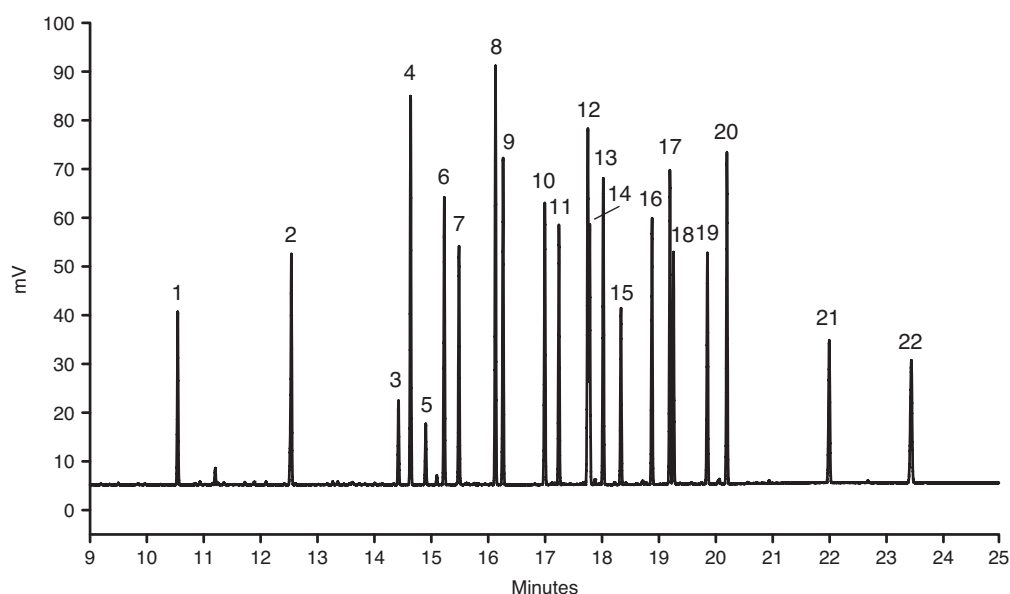


Figure 1: Chromatogram of 22 OPP standards at 20 µg/mL

Peak Number	Compound	t_r (min)	t_r %RSD (n=10)	Peak Area %RSD (n=10)
1	Dichlorvos	10.55	0.02	1.8
2	Mevinphos	12.55	0.02	2.0
3	Demeton O	14.43	0.02	2.6
4	Ethrophosphos	14.65	0.01	2.0
5	Naled	14.91	0.01	2.6
6	Phorate	15.24	0.01	1.8
7	Demeton S	15.50	0.01	1.9
8	Diazinon	16.14	0.01	1.9
9	Disulfoton	16.27	0.01	1.7
10	Methyl parathion	17.01	0.01	2.2
11	Fenchlorphos	17.26	0.01	2.0
12	Fenthion	17.77	0.02	2.3
13	Chlorpyrifos	17.80	0.02	3.4
14	Trichloronate	18.04	0.01	1.9
15	Merphos	18.35	0.01	1.9
16	Stirofos	18.90	0.01	2.0
17	Tokuthion	19.21	0.02	2.1
18	Impurity	19.27	0.01	2.2
19	Fensulfothion	19.87	0.01	2.1
20	Bolstar	20.22	0.01	2.0
21	Azinphos methyl	22.02	0.01	2.3
22	Coumaphos	23.46	0.01	2.1

Table 1: List of OPPs and their retention times peak area reproducibility

Conclusion

The TraceGOLD TG-5MS column and the deactivated, splitless quartz liner with quartz wool, when used in a TRACE 1310 GC instrument, demonstrated excellent performance for the separation and analysis of organophosphorus compounds with excellent peak shape, resolution, and reproducibility.

Reference

US EPA 8141B: <http://water.epa.gov/scitech/methods/cwa/index.cfm>

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