Waters™

Nota applicativa

Certified Sep-Pak Florisil for Sample Cleanup Organochlorine Pesticides and PCBs

Michael S. Young

Waters Corporation

Abstract

Florisil is often used for cleanup of soil extracts for pesticide analysis. A typical cleanup of a pesticide spiked soil extract was carried out using Certified Sep-Pak Florisil which was compared with a competitor Florisil cartridge. The competitor cartridge extract shows significant extractable interferences highly detrimental to the analysis. In contrast, the extract produced using the Certified Sep-Pak Florisil cartridge clearly shows significantly lower extractables leached from the sorbent or cartridge resulting in a much cleaner and more reliable analysis.

Introduction

Florisil is often used for cleanup of soil extracts for pesticide analysis. Florisil, a form of magnesium silicate, is a normal-phase sorbent and is used to remove polar interferences from the extract prior to GC or GC-MS analysis. In this SPE cleanup, the pesticide analytes are retained and concentrated on the cartridge; the analytes are then eluted with a weak solvent while polar interferences remain on the cartridge. Of primary importance is the cleanliness of the cartridge. The introduction of any interfering compounds resulting from leaching of extractables from the cartridge defeats the purpose of the cleanup. Figure 1 shows SPE protocols for cleanup of soil extracts for determination of pesticides and PCBs. Figure 2 presents GC-MS chromatograms that show typical cleanup of a pesticide spiked soil extract using Certified Sep-Pak Florisil compared with a competitor Florisil cartridge.

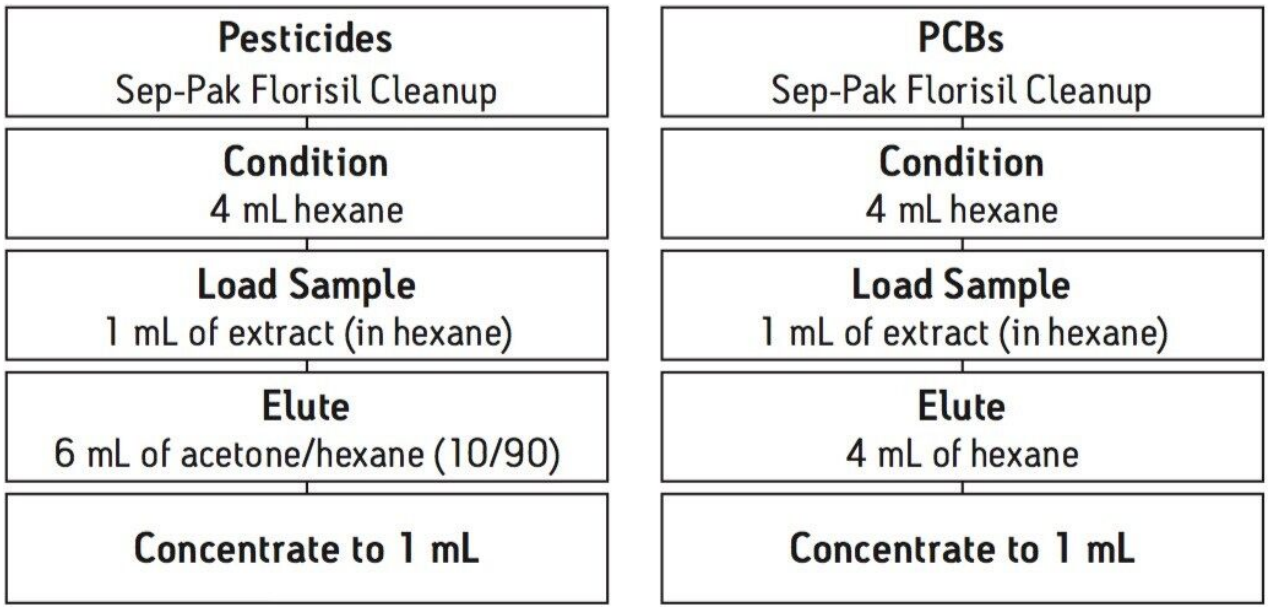

Figure 1. Certified Sep-Pak Florisil SPE protocols used in this study. Protocol 1 is for isolation or organochlorine pesticides from soil extract and protocol 2 is for isolation of PCBs from soil extract.

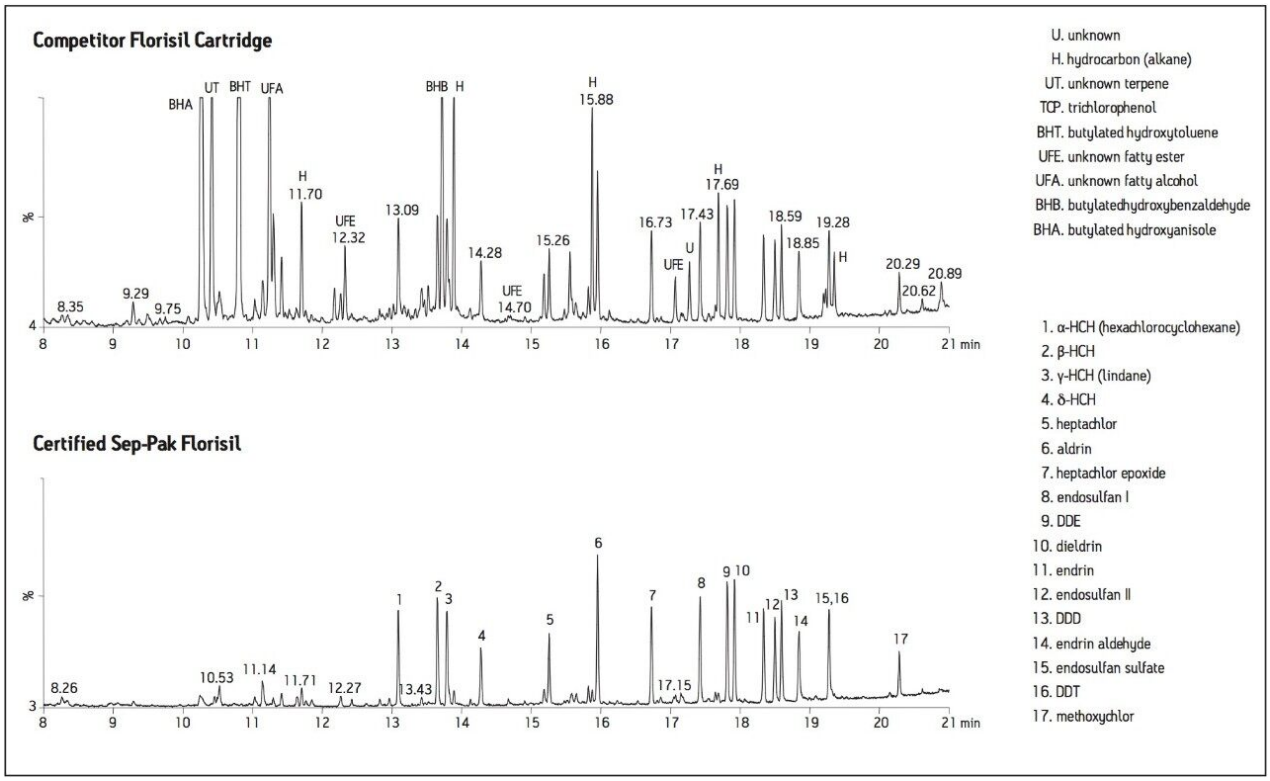

Figure 2. GC-MS analysis of organochlorine pesticides in soil extract using Certified Sep-Pak Florisil.

Experimental

Certified Sep-Pak Florisil Performance Verification

An analytical test was performed on a Certified Sep-Pak Florisil cartridge to verify the effectiveness of the device for removal of a standard polar interference from a soil extract with concomitant high recovery of target pesticides. For this test, trichlorophenol (TCP) is employed as the polar probe compound and the SPE protocol is the same as given in Figure 1. The sorbent should retain the TCP when the pesticides elute from the cartridge. This example demonstrates the performance of a Certified Sep-Pak Florisil cartridge for the cleanup of organochlorine pesticides at a 1 µg/kg (PPB) level in soil with GC-MS analysis. The results show excellent recovery of the pesticides with almost complete removal of the PCP from the extract. These results are summarized in Table 1.

Pesticide Recovery (n = 3)		
Compound	**Quant Ion**	**% Recovery (% RSD)**
α-HCH	181	104 (5.0)
β-HCH	181	103 (1.5)
γ-HCH (lindane)	181	104 (6.3)
δ-HCH	181	107 (8.7)
Heptachlor	100	98 (5.3)
Aldrin	263	97 (4.3)
Heptachlor epoxide	353	100 (2.1)
Endosulfan I	170	101 (3.0)
DDE	246	109 (4.8)
Dieldrin	263	102 (3.9)
Endrin	263	115 (2.3)
DDD	235	108 (0.9)
Endrin aldehyde	67	111 (9.4)
Endosulfan sulfate	272	96 (3.7)
DDT	235	105 (0.9)
Methoxychlor	227	109 (7.3)

TCP Removal		
Compound	Quant Ion	% Recovery (% RSD)
1. TCP	196	96.8

Table 1. Certified Sep-Pak Florisil performance verification by GC-MS

Determination of PCBs in Soil; Solvent Extraction and Solvent Exchange

A 5 g sample of soil was spiked with 10 ppb each of Arochlor 1016 and 1260 and placed in a 50 mL screwcap teflon extraction tube. To this sample, 10 g of anhydrous sodium sulfate and 30 mL of methylene choride (DCM) were added. The sample was placed on a rotory shaker for a 4 hour period. After that time the sample along with ca. 20 mL of DCM washing was filtered into a concentrator tube and evaporated to 1 mL using a TurboVap concentrator. 10 mL of hexane was added and the extract was again concentrated to 1 mL. This extract was used for SPE cleanup.

Determination of PCBs in Soil; Certified Sep-Pak Florisil Cleanup

Florisil is used to provide cleanup of soil samples for PCB analysis using GC with electron-capture detection (GC-ECD). This cleanup reduces the number of co-eluting peaks in the chromatographic analysis. Perhaps more importantly, the cleanup significantly reduces co-extractable substances from the final extract. These substances can contaminate GC columns and injection liners and profoundly affect the performance of the GC. Figure 3 shows chromatograms obtained from a solvent extract of soil spiked with 10 ppb of Aroclor 1016 and 1260. The lower chromatogram was obtained using no SPE cleanup and the upper chromatogram was obtained using a Certified Sep-Pak Florisil cartridge (SPE protocol for PCBs given in Figure 1). The SPE cleanup resulted in a cleaner baseline and an overall cleaner sample. Alongside each chromatogram is a photo of the actual sample extracts in GC vials; clearly, significant cleanup was achieved using the Certified Sep-Pak Florisil cartridge.

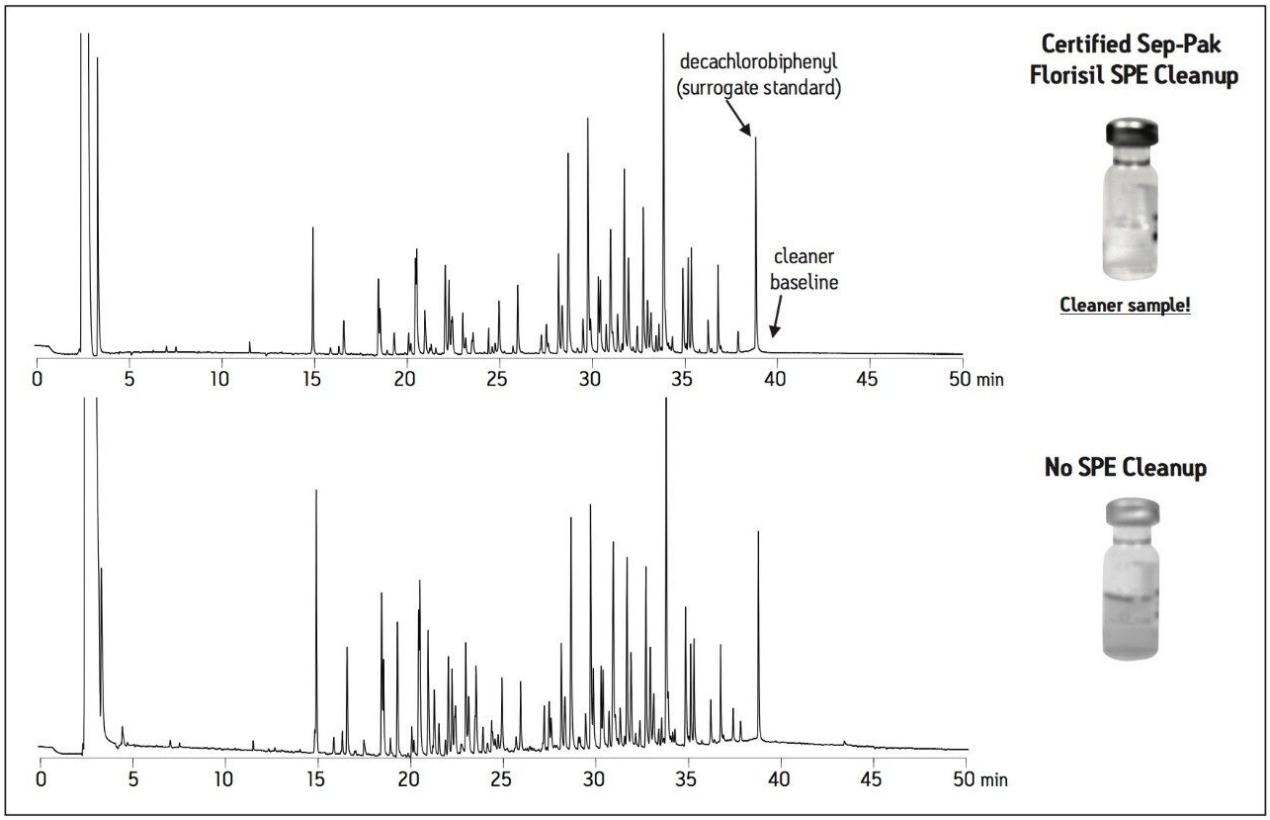

Figure 3. GC-ECD analysis of PCBs (10 ppb Arochlor 1016/1260) in soil extract using Certified Sep-Pak Florisil.

Results and Discussion

The competitor cartridge extract shows significant extractable interferences highly detrimental to the analysis. Among the compounds leached into the extract are preservatives (such as BHT) and various hydrocarbons related to the polymeric materials used to manufacture the cartridge. In contrast, the extract produced using the Certified Sep-Pak Florisil cartridge clearly shows significantly lower extractables leached from the sorbent or cartridge resulting in a much cleaner and more reliable analysis.

Conclusion

- Certified Sep-Pak Florisil cartridges are manufactured using strict performance and cleanliness specifications and QC-tested for extractables and leachables.
- Certified Sep-Pak Florisil cartridges provide increased sensitivity by elimination of contaminants introduced by the cartridge hardware and sorbents.
- Certified Sep-Pak Florisil cartridges are ideally suited for cleanup of soil extracts for determination of organochlorine pesticides and PCBs GC-MS or GC-FID.

Featured Products

720003169, March 2013

©2019 Waters Corporation. All Rights Reserved.