

# Optimizing Extraction of Multianalyte Suites from Water Samples Using Layered Solid Phase Extraction Columns

This innovative SPE technique is used in applications where multiple suites of analytes with a broad polarity range are to be extracted simultaneously from a single water sample, such as organochlorine, triazine and organophosphate pesticides, or endocrine disrupters.

Samples containing multiple analyte suites of very different chemical characteristics can be efficiently extracted using layered columns. This means that a single sample can be collected and extracted in order to determine multiple analyte suites, whereas separate samples would be needed if more than one extraction column was required. The layered column approach can significantly reduce costs and improve productivity.

Depending on the polarity range of analytes to be extracted, columns with different layers are available.



- » Extract multiple suites with a single SPE column
- » Improve productivity
- » Reduce costs

## The Problem

Large, non-polar analytes such as some chlorinated pesticides, PAHs and PCBs can often be retained from aqueous samples using ISOLUTE® C2 or C8 ('weakly retentive') sorbents.

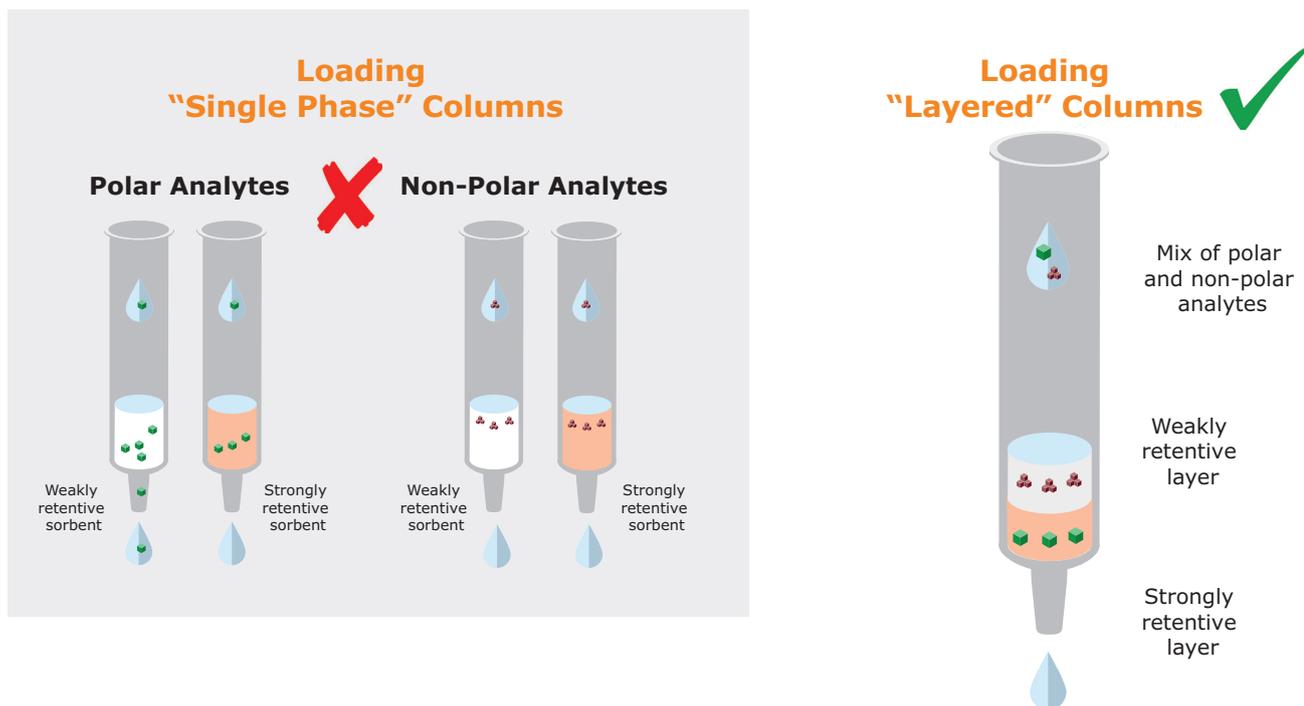
Smaller, more polar analytes such as acid herbicides often require much more retentive sorbents such as ISOLUTE C18 or ENV+ for efficient extraction from aqueous samples.

But, if the more retentive sorbent is used to extract the large, non-polar analytes, they will be retained very strongly, and may be difficult to elute (requiring large elution volumes).

## The Solution

Layers of sorbent with different retention and elution characteristics can be combined in a single SPE column to ensure efficient retention of analytes with a range of polarity/solubility.

When a less retentive sorbent is layered above a more retentive sorbent, the large, non-polar analytes will be retained on the top layer, while analytes requiring a more retentive phase will be retained on the bottom layer. This is illustrated in Figure 1.



**Figure 1:** When polar species are loaded onto a single phase column containing a 'weakly retentive' sorbent, they can breakthrough during load, ultimately leading to lower recoveries. When loading a mix of polar and non-polar analytes onto layered columns, non-polar species are trapped on the upper layer, and any more polar species that are not retained on the top layer will be trapped on the lower layer.

During elution, the larger, non-polar species are easily eluted from the less retentive upper layer, and are carried through the more retentive lower layer in the elution solvent without getting trapped. If these were retained on a single layered column containing the more retentive phase only, they would be more difficult to elute and require the use of larger volumes of elution solvent.

More polar, water soluble species are efficiently eluted from the lower layer together with the non-polar species, as the elution solvent passes through. This is illustrated in figure 2.

## Example Application

The layered column approach was applied to the extraction of a broad range of organic pollutants from drinking water, including acid herbicides, base-neutral herbicides, PAHs, phthalate esters and phenolic compounds<sup>1</sup>.

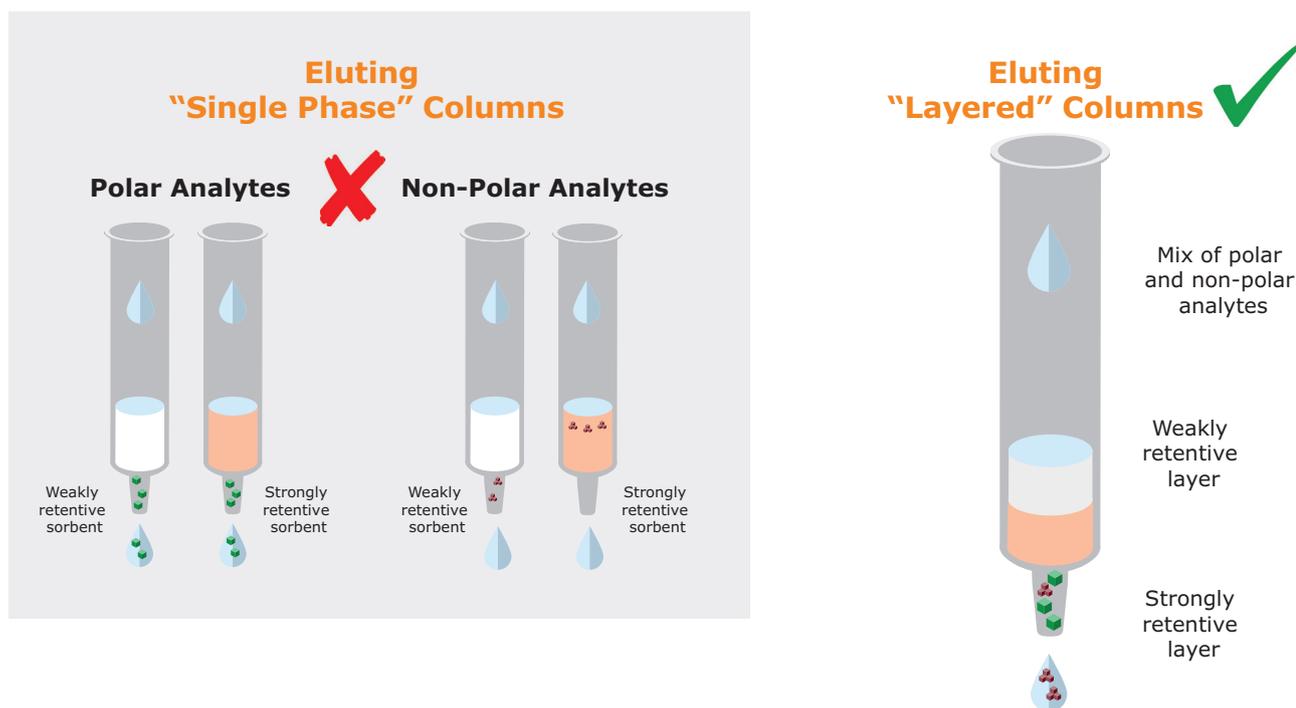
## Extraction Method

### SPE Column: ISOLUTE® C8/ENV+ Layered Column

In this column, the less retentive ISOLUTE® C8 upper layer efficiently retains non-polar analytes such as large PAHs. The more retentive ISOLUTE ENV+ layer retains polar species such as phenols and acid herbicides.

<b>Sample Pre-treatment</b>	Acidify sample to pH 2 with HCl. Add methanol (1%, v/v)
<b>Column Conditioning</b>	Methanol
<b>Column Equilibration</b>	Water
<b>Sample Load</b>	Up to 60 mL/min
<b>Dry Column for 10 mins</b>	
<b>Analyte Elution</b>	Acetone/ethyl acetate (1:1, v/v)

Following elution, the extract was evaporated to dryness and reconstituted in a solvent suitable for analysis. Each compound class was analysed separately, using optimized conditions.



**Figure 2:** Non polar analytes can be difficult to elute from strongly retentive single phase columns. When eluting a mix of polar and non-polar analytes from a layered column, a suitable elution solvent can be used to elute the whole range of analytes in a small volume.

## Typical Analyte Recovery

Analyte	% Recovery
<b>Base-neutral Herbicides</b>	
Metribuzin	90
Atrazine	92
Desethyl atrazine	89
Ametrine	90
Desisopropyl atrazine	85
Terbutaline	95
Arachlor	82
Metolachlor	95
Propachlor	80
Molinate	75
Propanil	75
Oxidiazon	85
Thiobencarb	75
<b>Acid Herbicides</b>	
2,4-D	86
MCPA	95
MCPB	94
MCPB	92
Bentazone	88
<b>Phthalate Esters</b>	
Diethylhexyl phthalate	92
Dibutyl phthalate	95
Benzylbutyl phthalate	96
Diethyl phthalate	98
Dimethyl phthalate	98
Diocetyl phthalate	94
<b>Phenols</b>	
Phenol	84
2-chlorophenol	90

Analyte	% Recovery
2-methylphenol	96
3-methylphenol	96
4-methylphenol	96
2-nitrophenol	87
2,4-dimethylphenol	95
2,4-dichlorophenol	90
2,6-dichlorophenol	90
4-chloro-3-methylphenol	86
2,4,6-trichlorophenol	85
2,4-dinitrophenol	87
4-nitrophenol	90
4,6-dinitro-2-Methylphenol	90
Pentachlorophenol	85
<b>PAHs</b>	
Naphthalene	87
Acenaphthalene	90
Acenaphthene	90
Fluorene	92
Phenanthrene	94
Anthracene	95
Fluoranthene	95
Pyrene	96
Benzo(a)anthracene	96
Chrysene	96
Benzo(a)fluoranthene	98
Benzo(k)fluoranthene	95
Benzo(a)pyrene	95
Indeno(1,2,3 cd)pyrene	93
Dibenzo(a,h)anthracene	95
Benzo(ghi)perylene	96

## Layered Column Ordering Information

Depending on the components of the analyte suite, choose a column with the lower layer suitable for extraction of the most polar analytes.

For analyte suites containing mainly very non-polar analytes, with no polar analytes (such as acid herbicides), the ISOLUTE® C2/C18(EC) column is recommended. For suites that include analytes which do not retain well on C18 type sorbents, column configurations containing ISOLUTE ENV+ are recommended.

Part Number	Description	Quantity
933-0050-B	ISOLUTE C2/C18(EC) 500 mg/3 mL	50
933-0100-C	ISOLUTE C2/C18(EC) 1 g/6 mL	30
934-0040-C	ISOLUTE C8/ENV+ 400 mg/6 mL	30
935-0040-C	ISOLUTE C18/ENV+ 400 mg/6 mL	30

## References

1. Davi et al, Intern. J. Environ. Anal. Chem 74 (1-4) 155-166

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