

SOLID-PHASE EXTRACTION

Principles, Techniques, and Applications

edited by

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PREFACE: SPE IN PERSPECTIVE

Few agree on who first performed a solid-phase extraction (SPE), or produced a "SPE" cartridge.¹ Fewer still agree on the date of the first use of SPE for sample preparation. Indeed, the term "solid-phase extraction" was only invented some years after products to perform the technique became available. It is common to find authors of articles in peer-reviewed journals who state model numbers of liquid chromatography pumps, quote buffer pHs to two decimal places, give the address of the distribution company that sold the chromatography column, or even quote the column serial or batch number. And yet, when describing the critical sample preparation step, these same authors claim that "a solid-phase extraction column was used" — no mention of type, size, flow rates, sample work-up, solvents used, let alone discussion of how well it worked! Where are you, editors and referees?

Which brings us to why a book such as this is needed: While most technologies develop from the laboratories of well-placed academic champions, from which theory, applications and acronyms pour forth, this humble technique has had no such birth. Indeed it has had precious little nurturing during the two decades since the first commercially available, pre-packed devices were produced. It is hoped that this book will serve as a catalyst for unified study of this field, as well as a resource for users seeking to optimize their sample preparation step. This book lays a foundation for future users of this powerful and subtle technology. Because expertise is distributed widely in the academic world and because the needs of users vary depending on the type of sample being prepared, it has been necessary to draw from a broad range of scientists in order to present a balanced study of the whole field.

During the preparation of this book I have had many fascinating conversations with contributing authors. One of the contributors, Dr. Martha Wells of Tennessee Technological University, beautifully captured the fun that has been had with this technique: "Today, as we continue to apply and expand SPE applications in our laboratory, I am still amazed at how well this relatively simple procedure can fractionate and concentrate complex chemical mixtures into more manageable subsamples. I realize that I am privileged to have experienced this analytical procedure's growth from a fledgling industry to today's 'workhorse' technique for environmental laboratories as well as for clinical and pharmaceutical applications.

¹ However, it is commonly agreed that the first commercially successful product was the Sep-Pak™ produced by Waters Inc. based on a patent of McDonald, Vivilecchia and Lorenz, bearing the title "Triaxially Compressed Beds" — U.S. Patent #4,211,658 (1980).

"Housed on my office shelves are keepsakes from these fifteen years, devices collected that were used to conduct and market SPE on bonded silicas. A 1979 sample Sep-Pak™ cartridge, the oldest item in my collection, is marked 'Prototype Use Only.' What a long way we have come since then! My collection illustrates that promoters found unique approaches to SPE sales: a game board with holes punched to hold SPE cartridges banded in either black or red for a round of checkers, or SPE columns packaged with Kool-Aid™ and instructions for separating red and blue dyes from this drink."

I am extremely grateful to everyone who contributed to this book. Several authors soldiered on through broken bones, new babies and, in one case, the near loss of his house in a brush fire! The whole process has been a wonderful learning experience for me. I am also indebted to the many who would-have-liked-to-but-couldn't, as well as to those who offered advice and encouragement.

I would especially like to thank Paul Wynne for going far beyond his initial duties by proof-reading several chapters and offering advice and insight on several others. Thanks are due to the staff at Marcel Dekker Inc., especially Brian Black, Anita Lekhwani and Linda Schonberg for their patience and expertise, and to Arnie, Dave, Doreen, Gwen, Kevin, Hung, Rob and the Helpdesk groups at Harbor City and Middelburg -- a pleasure doing business with you and thanks for your comments on the manuscript. Lastly, to my wife and Odette (who was still a theoretical consideration when this book was conceived), whose support and love make it all worthwhile, thank you.

Nigel Simpson

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- II. Solid-Phase Extraction: What It Is and What It Does
 - A. The Basic Steps of a Solid-Phase Extraction
- III. A Brief History of Solid-Phase Extraction
 - A. The Objectives of Solid-Phase Extraction
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I. THE SAMPLE PREPARATION PROBLEM

We have a sample in front of us. It is of unknown composition, but we know that it is complex, containing anywhere from a few hundred to many thousand chemical components. It is of uncertain physical form — perhaps it looks like a liquid, but it has solid particles floating in it. We think that a particular compound is present in that sample, somewhere and in some form, but we don't know how much is present, nor what form it takes.

And we have in front of us an attractive chromatogram of the compound of interest, our analyte, with another related compound that we will use as our measuring stick. We want to use the conditions given in the chromatogram, but the caption tells us that these compounds were injected in pure organic solvent at a concentration far higher than they would exist in our sample. How do we get our sample to this desirable state?

II. SOLID-PHASE EXTRACTION: WHAT IT IS AND WHAT IT DOES

Solid-phase extraction (SPE) is one of various techniques available to an analyst to bridge the gap that exists between the sample collection and the analysis step. Filtration, homogenization, precipitation, chemical reaction, solvent exchange, concentration, matrix removal, solubilization — these are just a few of the available tools that may be used individually or in combination to get the sample into a form compatible with the analytical instrument required for analysis. Solid-phase extraction is seldom used without other sample preparation steps, such as dilution or pH adjustment. However, as you will see in this and subsequent chapters, the action of performing SPE often simultaneously completes several other preparation goals. Moreover, SPE has, in several creative ways, been coupled with an analytical technique or another preparation method to enhance the benefits of each separate technique.

A. THE BASIC STEPS OF A SOLID-PHASE EXTRACTION

The simple and familiar practice of liquid/liquid extraction (LLE) is an excellent starting point from which to interpret SPE. In LLE the sample is agitated in the presence of an extracting solvent that is not miscible with the sample. When the sample/solvent mixture has settled after agitation, two layers of liquids are visible, one of which will contain most of the compound we are extracting. The shaking action has ensured that all parts of the sample come into contact with the extracting solvent. Compounds from the sample may pass into this extracting solvent and, given time, an equilibrium will be established between the two liquid layers. The equilibrium is described by the partition coefficient for the analyte, which is simply the ratio of concentrations for the analyte in the two liquids. A very high partition coefficient means essentially all the compound of interest will migrate into the extracting phase; a low coefficient means very little of the compound of interest has moved into the extracting phase. For most liquid/liquid extractions, properly chosen conditions will result in most of the

analyte being found in the extracting solvent, implying that the partition coefficient has been maximized. This occurs when the analyte interacts better with the extracting solvent than with the sample matrix. In other words, the extracting solvent provides a better environment for the analyte. To complete our LLE we now separate the two liquid layers and keep one for further manipulation, such as concentration, using a rotary evaporator or a stream of dry nitrogen.

1. Retention

In place of an extraction solvent we shall substitute a solid surface. When our compound distributes between the liquid sample and the solid surface, either by simple adsorption to the surface or through penetration of the outer layer of molecules on that surface, an equilibrium is set up, just as it was for LLE. We can define that distribution by a coefficient, K_o , which indicates to us what fraction of the analyte has remained in solution and what fraction has adsorbed on or entered the solid phase. Strictly speaking, this distribution coefficient should be defined in terms of activities of the analyte in either phase. However, convenience dictates that concentrations are used and therefore

$$K_o = [\text{analyte}]_{\text{sorbent}} / [\text{analyte}]_{\text{sample}} \quad (1)$$

If this process occurs in a column packed with a sorbent into whose outer layer the compound distributes, then we are dealing with a system that is no longer a "batch" partition like LLE. Instead the process more closely parallels distillation and Equation 1 converts to

$$k = 1/(1+k') \text{ or } V_o/V_R \quad (2)$$

where V_o and V_R are the void volume or empty space in the column and the retention volume respectively. If our compound is to be entirely trapped on this solid surface the distribution coefficient will be very large. So large, in fact, that chromatographic extraction, in contrast to elution chromatography, is best described as a pseudo-equilibrium process. We call the process whereby the analyte is completely adsorbed on the solid surface **RETENTION**. Chapter 13 in this book, titled "Matrix Solid Phase Dispersion," describes a novel and highly effective technique for getting compounds in a solid sample to retain on a solid surface. However, most Solid-Phase Extractions simply require a liquid sample to be passed through a bed containing sorbent particles onto which the analytes will retain.

2. Elution

Unless we can find a technique for identifying and quantifying the compound of interest while it is retained on the solid surface, we must find a

way to remove and collect it. Part II of this text deals with some advanced and unusual techniques for removal from the sorbent and collection of our analytes, such as using a stream of supercritical gas to desorb the analytes. But in a typical solid-phase extraction a simple liquid does the job very well. When a liquid provides a more desirable environment for the analyte than the solid phase does, then the compound of interest is desorbed and can be collected in the liquid as it exits the SPE device. This is called ELUTION. It is characterized by a k' between the concentration of the analyte on the solid surface and in the eluting liquid that is very small.

One way to view the solid phase is as an intermediary between the sample and the elution solvent and this highlights a very important difference between LLE and SPE. An elution solvent may be used which is miscible with the sample in a solid-phase extraction, because the elution solvent and the sample never come into direct contact. Thus, our sample may be aqueous but our SPE elution solvent may be methanol, which is miscible in all proportions with water. Such a scheme, impossible in a LLE, is not only possible with SPE — it accounts for the majority of all solid-phase extractions!

3. Rinsing or Washing

During the retention step, many compounds in our complex sample may have been retained on the solid surface at the same time as our compound of interest. Likewise, at elution it is likely that some of these co-retained compounds will be eluted with our compound of interest. To minimize the interferences these undesirable compounds will create during the analysis stage, we may add one or more wash steps between retention and elution, to attempt to remove or rinse them out. Each wash step involves another distribution between the analyte and the co-retained species, the solid surface and the liquid that is passing over it. You will control each step by careful selection of the wash, elution, and sample loading conditions.

4. A Complete Solid-Phase Extraction

The entire process of the solid-phase extraction is represented in Figure 1. You can now begin to appreciate the potential complexity but also the power of SPE. Each of the steps shown can be controlled. Thus, you can select the sorbent type (the solid phase that does the extraction); you can manipulate the sample to enhance retention of one chemical species over another; you can select an elution liquid that has properties that are not just desirable to the compound of interest, but which may be convenient for your method of analysis or for subsequent sample handling; and in between

Sample matrix

Wash solvent

Elution solvent

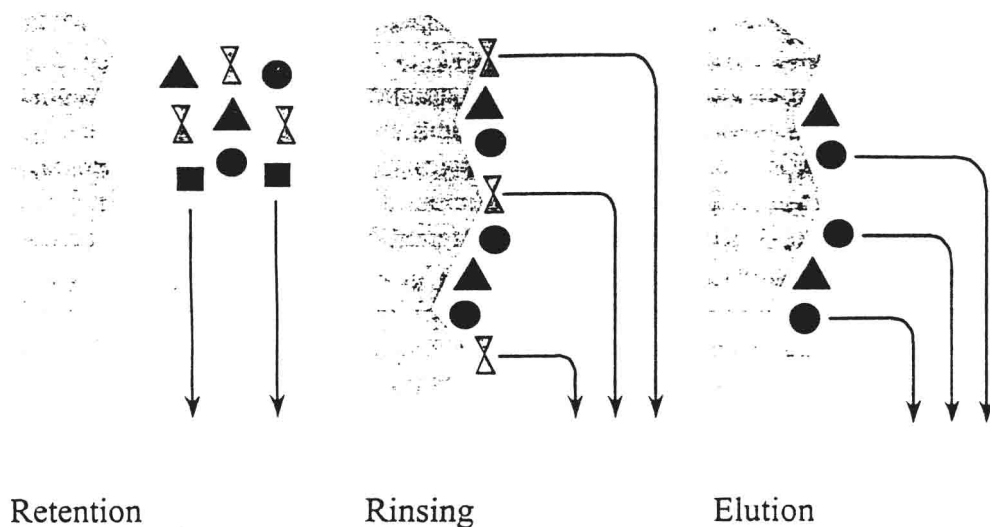


Figure 1. The three steps of a solid-phase extraction of a compound represented by ● after conditioning of the sorbent. A schematic view of what happens during sample loading, sorbent washing, and analyte elution. At each step some compounds will retain and others will be washed off or eluted.

you may use any number of wash steps to ensure that the final extract is of the desired purity.

III. A BRIEF HISTORY OF SOLID-PHASE EXTRACTION

Solid-phase extraction has been in use for thousands of years. Some scientists claim the first literature reference is to be found in the Bible, Exodus, Chapter 15, verses 24 and 25 to be exact (Riemon and Walton, 1970) even if the users were unaware of the science behind what they were doing! McDonald (1998) points out that the term is actually applied inaccurately since the extraction is not really performed by a solid phase but by a solid surface (adsorption), or at most a “meta-surface” like the organic layer of a C18 bonded silica. According to this strict interpretation, the extraction of volatile fragrances from rose petals by medieval laborers in Grasse, France, who performed the feat by embedding the petals in paraffin wax, counts as