Table 2 Concentration of water and difference in absorbance for cut-off (ΔA) of solvents before (C_b) and after (C_a) purification

Solvent	ΔA *(% v/v)*	C_b *(mg L^{-1})*	C_a *(mg L^{-1})*
Acetonitrile A	69.7	2216.0	167.0
Acetonitrile B	10.2	297.0	143.0
Benzene	24.6	308.0	27.5
Cyclohexane	16.8	49.0	4.5
n-Heptane	21.3	18.8	7.7
n-Hexane	17.2	19.6	8.6
Methanol A	77.05	650.0	134.0
Methanol B	3.6	120.0	114.0
Tetrahydrofuran	61.3	1081.0	32.6
Toluene	23.1	167.0	10.0

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methods have been utilized for measuring the characteristic parameters. Generally, chromatographic techniques such as gas chromatography (GC), LC, thin-layer chromatography (TLC) have been used, but ultraviolet (UV), infra-red (IR) and nuclear magnetic resonance (NMR) spectroscopy can also be applied. Water in many organic solvents is usually determined by Karl Fischer titration. On the basis of experimental data obtained before and after purification the efficiency of the clean-up procedure is determined. In general, the efficiency of purification process, e.g. the recovery, is expressed by the coefficient R. This parameter is defined as the ratio of the volume or concentration of removed impurities to the volume or concentration of solvent before purification:

$$R \pm \sigma = (V_a \pm \sigma_a)(V_b \pm \sigma_b) \times 100\% \text{ (v/v)} \quad [1]$$

or:

$$R \pm \sigma = (C_a \pm \sigma_a)(C_b \pm \sigma_b) \times 100\% \text{ (v/v)} \quad [2]$$

where V_a, C_a and V_b, C_b denote volume or concentration of the removed impurities and solvent samples, respectively, and $\sigma, \sigma_a, \sigma_b$ are individual standard deviations.

Table 2 summarizes the results of solvent purification by frontal analysis. In each case, purification of the solvent improves its absorbance at the cut-off point (ΔA).

The small improvement found in the case of the LC grade methanol B is as expected for this high-purity solvent. The high impurity (absorbance difference, $\Delta A = 77.5\%$) of analytical grade methanol A precludes its use in LC investigations. Similarly, toluene, benzene, tetrahydrofuran and acetonitrile A cannot be used in LC measurements without prior purification.

Recovery values for the purification of water, acetonitrile, alcohols, ketones, aliphatic and aromatic hydrocarbons obtained in distillation methods are usually in the range 85–95% (v/v). In the case of halogenated solvents this range is narrower, i.e. 75–80% (v/v). Utilizing membrane techniques for solvent clear-up, it is possible to obtain recovery in the range 90–97% (v/v).

See also: **II/Distillation:** Laboratory Scale Distillation. **Membrane Separations:** Filtration. **III/Flash Chromatography.**

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SORBENT SELECTION FOR SOLID-PHASE EXTRACTION

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Solid-phase extraction (SPE) is a method of sample preparation that concentrates and purifies analytes from solution by sorption onto a disposable solid-phase cartridge, followed by elution of the analyte

with a solvent appropriate for instrumental analysis. The mechanisms of retention include reversed phase, normal phase, size exclusion, and ion exchange. Solid-phase extraction was invented in the mid 1970s as an alternative approach to liquid–liquid extraction for sample preparation.

Initially, SPE was based on the use of polymeric sorbents, such as XAD resins, which were packed in small disposable columns for use on drug analysis. The early environmental applications consisted of both XAD resins and bonded-phase sorbents, such as C_{18}. These precolumns were used for sample trace enrichment prior to liquid chromatography and were often done on line, which means at the same time as liquid chromatography. However, these first, steel, on-line precolumns were quickly replaced with an off-line column made of plastic, in order to be both inexpensive and disposable. Eventually, the term solid-phase extraction was coined for these low-pressure extraction columns.

SPE columns are now typically constructed of polypropylene or polyethylene and filled with 40-μm packing material with different functional groups. A 20-μm polypropylene frit is used to contain from 50 mg to 10 g of packing material. A liquid sample is passed through the column and analytes are concentrated and purified. The sample volume that can be applied ranges from 1 mL to over 1 L. The sample may be applied to the column by positive pressure or by vacuum manifold. After quantitative sorption of the analyte, it is removed with an appropriate elution solvent.

Therefore, SPE is a form of 'digital' liquid chromatography that removes the solute onto a solid-phase sorbent by various sorption mechanisms. The term 'digital' refers to the on/off mechanism of sorption and desorption. The goal of SPE is to quantitatively remove the analyte from solution and completely recover it in an appropriate solvent. Purification consists of removing the analyte from interfering compounds and concentrating the analyte in a small volume of solvent. For example, pesticides are concentrated from a water sample by SPE into a small volume of organic solvent for analysis by gas chromatography/mass spectrometry. Interfering substances, such as humic and fulvic acids, ionic metabolites and salts are removed.

Typically, SPE replaces liquid–liquid extraction as a sample preparation tool and provides a method that is simple and safe to use. The benefits of SPE include: high recoveries of analytes; purified extracts; ease of automation; compatibility with chromatographic analysis; and reduction in the consumption of organic solvents. As a result of the flexibility that SPE offers, it has found application in the preparation of environmental, clinical, and pharmaceutical samples. The simplicity of the SPE procedure and the use of disposable SPE supplies have encouraged the design of automated sample preparation stations, which decrease the time and cost of sample preparation. Finally, recent advances in on-line methods of SPE allow automation of sample preparation directly to both liquid and gas chromatography.

How to do SPE

Figure 1 illustrates the four-step process of SPE. First the solid-phase sorbent is conditioned (step 1). This simply means that a solvent is passed through the sorbent to wet the packing material and to solvate the functional groups of the sorbent. Furthermore, the air that is present in the column is removed and the void spaces are filled with solvent. Typically, the conditioning solvent is methanol, which is then followed with water or an aqueous buffer. The methanol followed by water or buffer activates the column in order for the sorption mechanism to work properly for aqueous samples. Care must be taken not to allow the bonded-silica packing or the polymeric sorbent to go dry. In fact, if the sorbent dries for more than several minutes under vacuum, the sorbent *must* be reconditioned. If it is not reconditioned the mechanism of sorption will not work effectively and recoveries will be poor for the analyte.

Another cleaning step of the sorbent may also be added during conditioning, if necessary. Simply, the eluting solvent is passed through the column after the methanol wetting step to remove any impurities that may be present in the packing material. This cleaning step would then be followed by methanol and aqueous buffer, which prepares the column for sample addition.

Next, the sample and analyte are applied to the column (step 2, Figure 1). This is the retention or loading step. Depending on the type of sample, from 1 mL to 1 L of sample may be applied to the column either by gravity feed, pumping, aspirated by vacuum, or by an automated system. It is important that the mechanism of retention holds the analyte on the column while the sample is added. The mechanisms of retention include Van der Waals (also called nonpolar, hydrophobic, partitioning, or reversed-phase) interaction, hydrogen bonding, dipole–dipole forces, size exclusion, and cation and anion exchange. During this retention step, the analyte is concentrated on the sorbent. Some of the matrix components may also be retained and others may pass through, which gives some purification of the analyte.

Step 3 (Figure 1) is to rinse the column of interferences and to retain the analyte. This rinse will

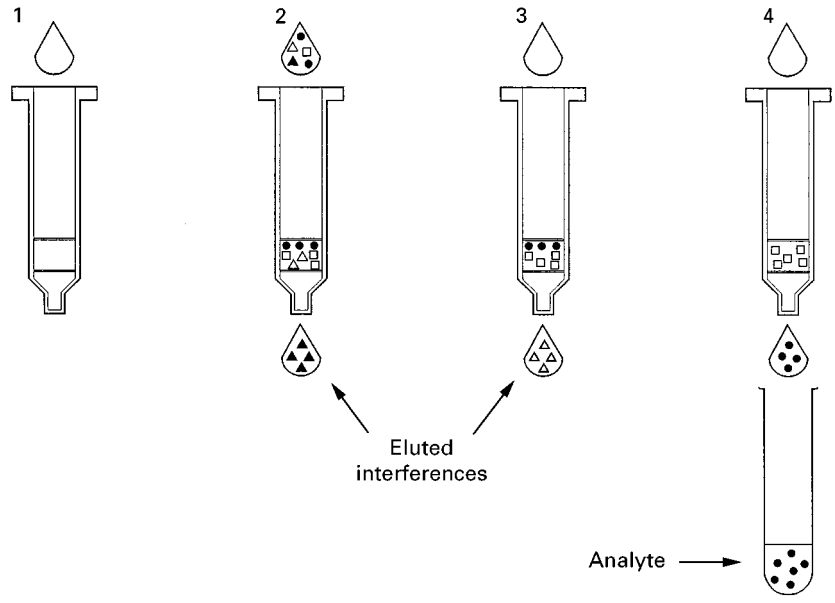

Figure 1 The four-step process of solid-phase extraction. Step 1, condition sorbent; step 2, apply sample and analyte; step 3, wash; step 4, analyte elution. (Reprinted from Thurman and Mills (1998) Copyright John Wiley & Sons, Inc.)

remove the sample matrix from the interstitial spaces of the column, while retaining the analyte. If the sample matrix is aqueous, an aqueous buffer or a water/organic-solvent mixture may be used. If the sample is dissolved in an organic solvent, the rinse solvent could be the same solvent.

Finally, in step 4 an appropriate solvent is used that is specifically chosen to disrupt the analyte–sorbent interaction, resulting in elution of the analyte from the sorbent. The eluting solvent should remove as little as possible of the other substances sorbed on the column. This is the basic method of solid-phase extraction.

Columns and Apparatus for SPE

The sorbents used for SPE are packaged in three basic formats. There are discs, cartridges, and syringe barrels. **Figure 2** shows the different types of presentation of SPE products. The discs are available in different diameters from 4 to 90 mm, the 'standard' disc size being 47 mm. Cartridges vary from as little as 100 mg to 1 g or more. Syringe barrels are available in different volumes and with different masses of packing material. Syringe barrels range in size from 1 to 25 mL and packing weights from 50 mg to 10 g. These various sorbents allow for the effective treat-

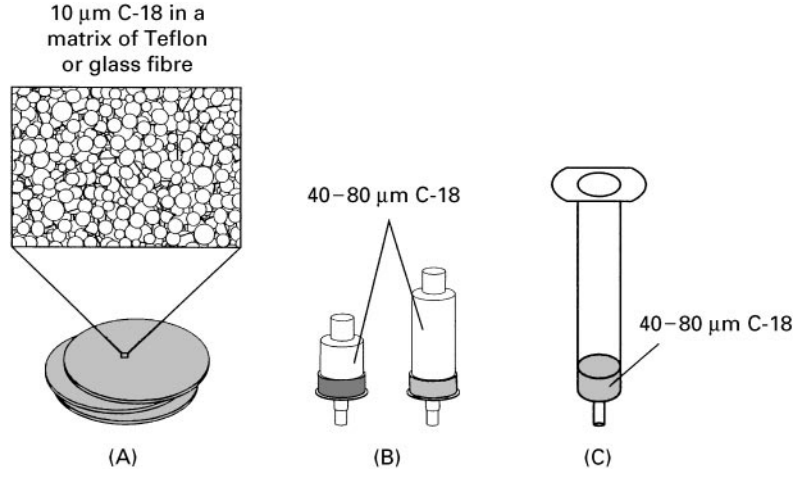

Figure 2 The three formats of SPE: (A) discs; (B) cartridges; and (C) syringe barrels. (Reprinted from Thurman and Mills (1998) Copyright John Wiley & Sons, Inc.)

ment of different types of sample and different sample volumes.

Currently, the most commonly used format for SPE consists of a syringe barrel that contains 40-μm sorbent material, with a 20-μm polypropylene frit at the bottom and a 20-μm polypropylene frit at the top of the syringe. The syringe barrel is typically polypropylene with a male Luer-tip fitting and is disposable. Some vendors do make glass syringe barrels and Teflon frits, but these configurations are used less frequently. The glass and Teflon system is used when one is interested in the analysis of plasticizers or is concerned with the potential sorption of specific analytes onto the polyethylene tube.

Solvent reservoirs may be used to increase the volume of the syringe barrel. Reservoirs are typically 50–100 mL in volume. Coupling fittings are used to join the reservoirs and syringe barrels between the Luer fitting and the opening of the syringe barrel (**Figure 3**).

The barrel of the syringe terminates in a male Luer tip. The male Luer tip is the standard fitting on SPE cartridges, so that they are interchangeable with different SPE vacuum manifolds. The vacuum manifold is used to draw the sample and eluting solvents through the syringe barrel under negative pressure by applying a vacuum to the manifold. **Figure 4A** shows a typical vacuum-manifold system, which is fitted with a small vacuum pump and a waste receiver. Stopcock valves are available to control the vacuum applied to each column. Other types of sample processing that may be used include centrifugation (Figure 4C) and positive pressure (Figure 4D), which forces the sample through the syringe barrel from above. Simple gravity flow through the syringe barrel or cartridge may also be used (Figure 4B).

A typical solid-phase extraction cartridge (see Figure 2) consists of a polyethylene body with both a female and male Luer tip for positive pressure from a syringe, or negative pressure from a vacuum manifold. Polyethylene frits measuring 20 μm are placed at either end of the cartridge to hold the packing material in place. The packing material is packed and compressed to improve or optimize flow characteristics.

The third type of SPE format is the disc, which is available in several styles by different manufacturers. One of the most popular extraction discs is the Empore® extraction disc, which consists of 8–12 μm particles of packing material embedded in an inert matrix of polytetrafluoroethylene (PTFE) fibrils. Because the particles are suspended in PTFE, no binder is required to give structure to the disc and the matrix is essentially inert. The discs are not coated with PTFE so that they can interact with the solvent and sample during extraction. The discs are available in a membrane format as loose discs, or are placed in a syringe-barrel format called an extraction disc cartridge. The syringe-barrel format consists of a standard polyethylene syringe that is fitted with a 20-μm Teflon frit, an Empore® disc, and a prefilter of glass fibre. This arrangement allows for micro-scale work using the disc. Discs are conditioned and used in

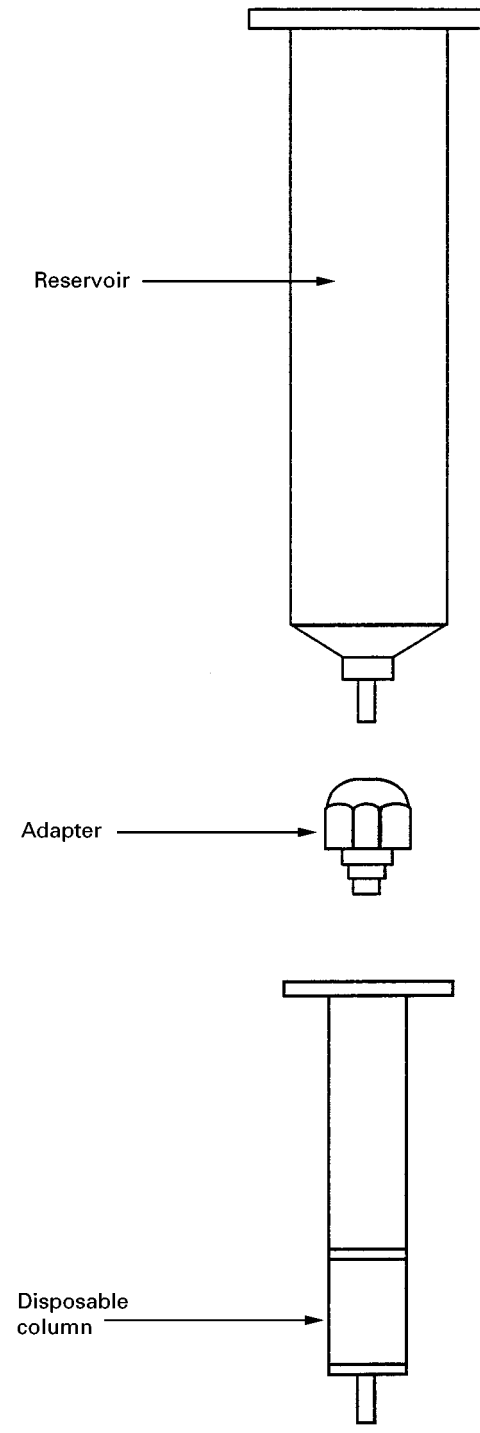

Figure 3 Disposable column and reservoir. (Reprinted from Thurman and Mills (1998) Copyright John Wiley & Sons, Inc.)

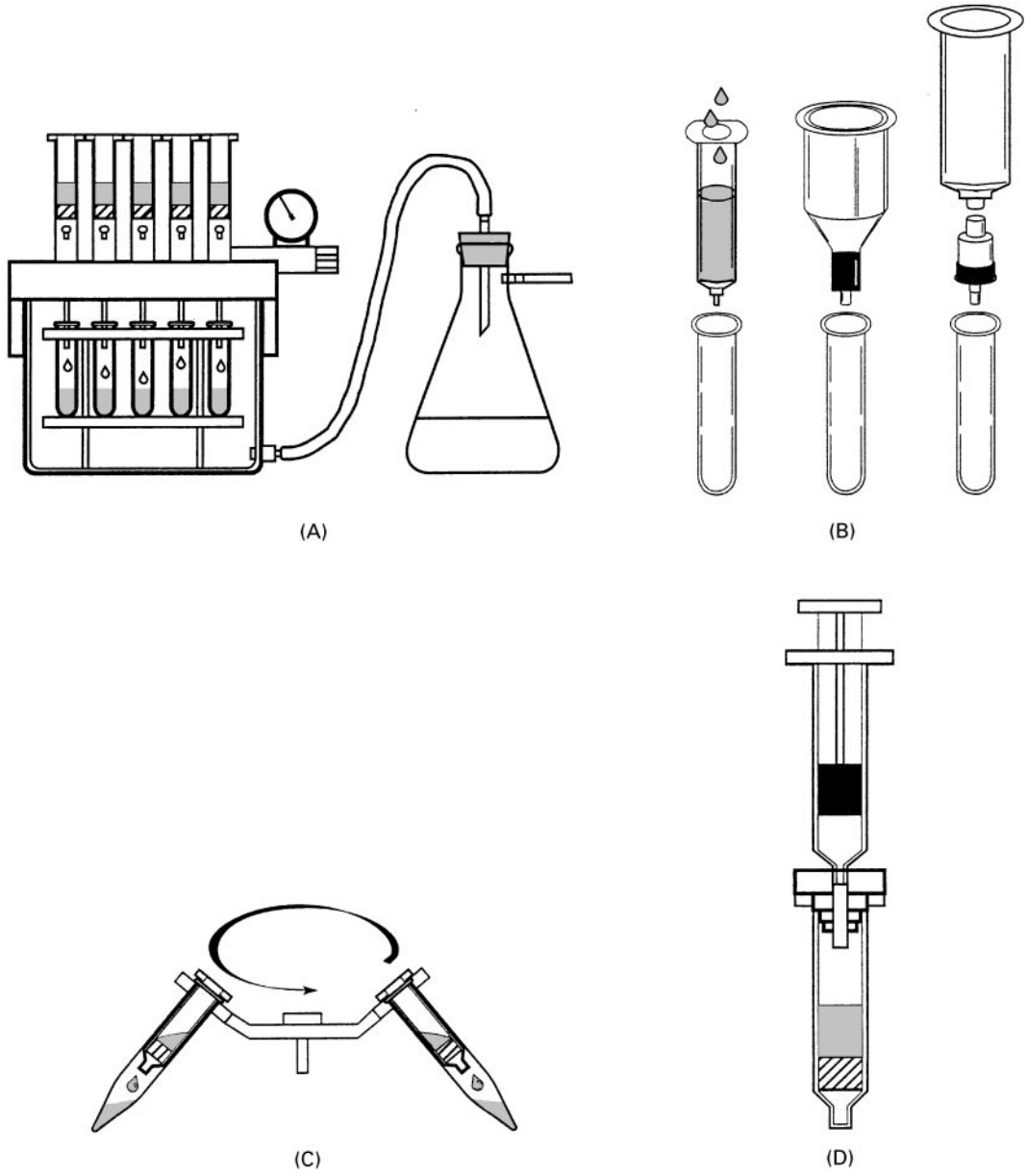

Figure 4 Techniques for processing SPE cartridges: (A) vacuum manifold; (B) gravity; (C) centrifugation; and (D) positive pressure. (Reprinted from Thurman and Mills (1998) Copyright John Wiley & Sons, Inc.)

a similar fashion to the packed columns, with flow of sample by negative pressure by vacuum.

A major advantage of the disc format is rapid mass transfer because of the greater surface area of the 8–12 μm particles, which results in high flow rates for large volume samples. This rapid flow rate is especially useful for environmental samples where 1 L of water may be processed in as little as 15 min. Rapid mass transfer owing to embedding of small particles into the disc also means that channelling is reduced and small volumes of conditioning and elution solvents may be used. For example, a 4-mm disc in syringe format (extraction disk cartridge) requires only 100 μL of elution solvent, and a 7-mm disc uses only 250 μL. This volume is small compared with the millilitre amounts applied to a 3–5-mL syringe barrel that contains loose packing.

Another type of disc called SPEC® manufactured by Ansys, Inc., uses a glass-fibre matrix rather than Teflon to hold the sorbent particles. This disc has a somewhat more rapid flow rate and is more rigid and thicker than the Teflon disc. There is another disc called the Speedisk® manufactured by J. T. Baker, which consists of 10-μm packing material that is sandwiched between two glass-fibre filters without any type of Teflon binder.

Sorbents and Modes of Interaction

The sorbents used for SPE are similar to those used in liquid chromatography, including normal phase, reversed phase, size exclusion, and ion exchange. Normal-phase sorbents consist of a stationary phase that is more polar than the solvent or sample matrix that is applied to the SPE sorbent. This means that water is not usually a solvent in normal-phase SPE because it is too polar. Normal-phase sorbents, therefore, are used in SPE when the sample is an organic solvent containing an analyte of interest. Polar interactions, such as hydrogen bonding and dipole–dipole interactions, are the primary mechanisms for solute retention.

Reversed-phase sorbents are packing materials that are more hydrophobic than the sample. Reversed-phase sorbents are commonly used in SPE when aqueous samples are involved. The mechanism of interaction is Van der Waals forces (also called non-polar, hydrophobic, or reversed-phase interactions) and occasionally secondary interactions such as hydrogen bonding and dipole–dipole interactions. Size-exclusion sorbents utilize a separation mechanism based on the molecular size of the analyte. It is a method only recently being used in SPE, usually in conjunction with reversed phase or ion exchange. Ion-exchange sorbents isolate analytes based on the ionic state of the molecule, either cationic or anionic, where the charged analyte exchanges for another charged analyte that is already sorbed to the ion-exchange resin. SPE applications in this case are essentially identical to classical ion exchange.

Thus, the mechanisms of interaction include: hydrogen bonding and dipole–dipole forces (polar interactions); Van der Waals forces (non-polar or hydrophobic interactions); size exclusion; and cation and anion exchange. Some sorbents combine several interactions for greater selectivity. The extensive line of sorbent chemical structures facilitates one of the most powerful aspects of SPE, which is selectivity. Selectivity is the degree to which an extraction technique can separate the analyte from interferences in the original sample. The number of possible interactions between the analyte and the solid phase facilitates this selectivity.

Table 1 lists the common sorbents that are available for SPE and their mode of action (i.e. reversed phase, normal phase, ion exchange, and size exclusion). Typically the sorbents consist of 40-μm silica gel with approximately 60-Å-pore diameters. Chemically bonded to the silica gel are the phases for each mode of action. For reversed-phase sorbents, an octadecyl (C_{18}), octyl (C_8), ethyl (C_2), cyclohexyl, and phenyl functional groups are bonded to the silica. Typical loading of reversed-phase sorbents varies from approximately 5% for the C_2 phase to as much as 17% for the C_{18} phase. The per cent loading is the

Table 1 Common sorbents available for SPE

Sorbent	*Structure*	*Typical loading*
Reversed phase		
Octadecyl (C_{18})	$-(CH_2)_{17}CH_3$	17%C
Octyl (C_8)	$-(CH_2)_7CH_3$	14%C
Ethyl (C_2)	$-CH_2$-CH_3	4.8%C
Cyclohexyl	$-CH_2CH_2$-cyclohexyl	12%C
Phenyl	$-CH_2CH_2CH_2$-phenyl	10.6%C
Graphitized carbon	Aromatic carbon throughout	
Copolymers	Styrene divinylbenzene	
Normal phase		
Cyano (CN)	$-(CH_2)_3CN$	10.5%C, 2.4%N
Amino (NH_2)	$-(CH_2)_3NH_2$	6.4%C, 2.2%N
Diol (COHCOH)	$-(CH_2)_3OCH_2CH(OH)CH_2(OH)$	8.6%C
Silica gel	–SiOH	—
Florisil	Mg_2SiO_3	—
Alumina	Al_2O_3	—
Ion exchangers		
Amino (NH_2)	$-(CH_2)_3NH_2$	1.6 meq g^{-1}
Quaternary amine	$-(CH_2)_3N^+(CH_3)_3$	0.7 meq g^{-1}
Carboxylic acid	$-(CH_2)_2COOH$	0.4 meq g^{-1}
Aromatic sulfonic acid	$-(CH_2)_3$-phenyl-SO_3H	1.0 meq g^{-1}
Size exclusion		
Wide-pore hydrophobic (butyl)	$-(CH_2)_3CH_3$	5.9%C
Wide-pore ion exchangers–COOH		12.2%C

amount of C_2 or C_{18} phase that is present by weight of carbon. The capacity of the sorbent in mg g^{-1} of analyte that may be sorbed is related to both the chemistry of the phase and the loading weight of carbon. Polymeric sorbents, such as styrene divinylbenzene and carbon, also are used for reversed-phase SPE. These sorbents were some of the classical reversed-phase sorbents introduced in the 1960s. They are currently produced in purified form and are useful for the isolation of more polar solutes that have low capacities on the C_{18} reversed-phase sorbents.

For normal-phase SPE, cyanopropyl (CN), aminopropyl (NH_2), and diol functional groups are chemically bonded to the silica gel. The loading on the cyano, amino, and diol columns are sufficiently large ($\sim$6–10% as carbon) in that they may sometimes be used for reversed-phase applications, especially for the removal of hydrophobic solutes from water or other polar solvents. These hydrophobic solutes would otherwise sorb too strongly to a more hydrophobic C_8 or C_{18} sorbent and would be difficult to elute. Straight silica gel is also used for normal-phase SPE along with Florisil (magnesium silicate) and alumina (aluminum oxide in neutral, basic, and acidic forms).

Ion-exchange sorbents usually contain both weak and strong cation and anion functional groups bonded to the silica gel (Table 1). Strong cation-exchange sorbents contain ion-exchange sites consisting of sulfonic acid groups, and weak cation-exchange sorbents contain sites consisting of carboxylic acid groups. Strong anion-exchange sites are quaternary amines, and weak anion-exchange sites are primary, secondary, and tertiary amines. Strong and weak refers to the fact that strong sites are always present as ion-exchange sites at any pH, while weak sites are only ion-exchange sites at pH values greater or less than the p*K*a, which determines whether a site contains a proton or not. The typical loading for an ion-exchange sorbent is expressed in meq g^{-1} of sorbent, which is called the exchange capacity of the sorbent. The values vary from $\sim$0.5 meq g^{-1} to 1.5 meq g^{-1}. These exchange capacities are somewhat less than a typical ion-exchange resin, which will have from 2 to 5 meq g^{-1} because of a higher density of ion-exchange sites. Also these SPE ion-exchange sorbents are not as rugged as the polymeric ion-exchange resins because of the silica matrix of the SPE sorbent, which is susceptible to dissolution by strong acid or base. The typical ion-exchange resin, however, consists of a cross-linked styrene-divinylbenzene polymer.

Size-exclusion sorbents, called wide-pore sorbents (Table 1), use a silica-gel matrix with a large pore size (approximately 275–300 Å) rather than the 60-Å pores of most bonded-phase silicas. The advantage of the larger pore size is that molecules of larger molecular weight ($>$2000 daltons) may enter the pore of the sorbent and sorb by hydrophobic, polar, or ion exchange interactions. Two examples are shown in Table 1. One is a hydrophobic sorbent of C_4 (butyl) with a carbon loading of almost 6%, and the other is a weak cation sorbent using the carboxyl exchange site.

Another packing material, which is not listed in Table 1, was recently introduced for drug analysis. It is a mixed-mode resin. This packing material contains both a bonded reversed-phase group (typically a C_8) and a cation-exchange group on a silica gel or a polymeric matrix. The combination of bonded groups is used so that both types of mechanisms retain the analyte at different times, or simultaneously, in the clean up of complex samples of urine and blood. The principle of the mixed-mode resin is that different wash solvents may be used to remove interferences, but that the solute is always retained by one or both of the interactions.

Applications of SPE

Table 2 shows a general application guide for the use of SPE sorbents. The C_{18} reversed-phase sorbent has historically been the most popular packing material and has been used most frequently. The surface of the sorbent is one of the most hydrophobic and has a large capacity. Capacity is the amount of analyte sorbed (usually expressed in mg g^{-1}) before breakthrough occurs. Applications of C_{18} reversed phase include: isolation of hydrophobic species from aqueous solutions, such as drugs and metabolites from urine, serum, plasma, and other biological fluids; desalting of peptides and oligonucleotides; isolation of pigments from wine and beverages; and trace enrichment of pesticides from water for analysis by gas chromatography/mass spectrometry or high pressure liquid chromatography.

Graphitized carbon and reversed-phase polymeric sorbents are also frequently used in environmental applications, such as trace enrichment, for soluble molecules that are not isolated by reversed-phase sorbents, such as C_{18}. Water soluble analytes require a more hydrophobic sorbent with greater surface area per gram for complete retention. Carbon and polymeric sorbents may also be used for polar metabolites of drugs and pharmaceuticals that are poorly retained on C_{18}. Another advantage of the aromatic sorbents is their selective interaction with the aromatic rings of analytes. Because both the graphitized carbon and the styrene divinylbenzene structures contain aromatic rings, they have the ability to sorb analytes by a

Table 2 Selected application guide for SPE

Sorbent	*Application*
C_{18}	Reversed phase application one of the most hydrophobic phases Drugs in serum, plasma, and urine Organic acids in wine Pesticides in water by trace enrichment
Graphitized carbon polymeric sorbents (styrene divinylbenzene)	Reversed phase application one of the most hydrophobic phases Trace enrichment of polar pesticides from water Isolation of polar drug metabolites
C_8	Reversed phase application–hydrophobic phase Drugs from serum, urine, and plasma Peptides in serum and plasma
Silica	Normal phase application–polar neutral phase Isolation of low to moderate polarity species from non-aqueous solution Lipid classification
Florisil	Normal phase application–polar slightly basic phase Isolation of low to moderate polarity species from non-aqueous solution Pesticides in food and feeds Polychlorinated biphenyls is transformer oil
Alumina A	Normal phase application–acidic polar phase Isolation of hydrophilic species in non-aqueous solution Low capacity cation exchange
Cation exchange	Cation exchange phase Isolation of cationic analytes in aqueous or non-aqueous solutions Fractionation of weakly basic proteins and enzymes
Anion exchange	Anion exchange phase Isolation of anionic analytes in aqueous or non-aqueous solutions Extraction of acidic and weakly acidic proteins and enzymes
Mixed mode	Reverse phase (C_8) and cation exchange phase Isolation of basic and amphoteric drugs from serum, plasma, and urine
Aminopropyl NH_2	Normal phase, reverse phase, and weak cation exchange Low capacity weak anion exchanger Drugs and metabolites from body fluids Petroleum and oil fractionation
Cyanopropyl CN	Normal phase and reversed phase Analytes in aqueous or organic solvents Drugs and metabolites in physiological fluids
Diol OH	Normal phase and reversed phase Analytes in aqueous or organic solvents Drugs and metabolites in physiological fluids

specific pi-pi interaction. This sorption mechanism may selectively isolate aromatic compounds.

The C_8 reversed-phase sorbents (Table 2) are often the most popular sorbent for drug analysis because of a shorter hydrocarbon chain than a C_{18} sorbent. The shorter chain length makes it much more easy for secondary interactions between the analyte and the silica gel which enhances retention of the analyte. This added interaction is useful in the purification of drugs and metabolites from blood and urine because they contain basic nitrogen atoms that may hydrogen bond to the silica gel.

Normal-phase sorbents such as silica and Florisil are used to isolate low to moderate polarity species from non-aqueous solutions. Examples of applications include lipid classification, plant-pigment separations, and separations of fat-soluble vitamins from lipid extracts, as well as the clean up of organic solvent concentrates obtained from a previous SPE method or liquid–liquid extraction. Alumina is

used to remove polar species from non-aqueous solutions. Examples include vitamins in feeds and food, and antibiotics and other additives from feed. Normal-phase chromatography has been used for a number of years and most applications for normal-phase column chromatography may be easily transferred over to normal-phase SPE.

Cation and anion exchange is used to isolate ionic compounds from either aqueous or non-aqueous solutions. Examples of applications are: isolation of weakly basic proteins; removal of acidic pigments from wines and fruit juices; and the removal of organic acids from water. Many of the applications of classical ion exchange may be used in ion-exchange SPE; however, care must be exercised in the use of strong acids and bases with SPE ion-exchange sorbents that are based on a silica matrix. Furthermore, care must be taken not to exceed the ion-exchange capacity of the sorbent.

Finally, sorbents such as aminopropyl, cyanopropyl, and diol can be used for both reversed-phase and normal-phase separations. Many manufacturers supply their sorbents in variety packs, which may be used for methods development. Also quality assurance reports are commonly available for the various sorbents, which is a good indication of their reproducibility.

Automation of SPE

Automation of a manual SPE method can provide many benefits, which include safety, improved results, and cost savings. Because automated workstations are mechanical they can operate in environments that are hostile, for example, noisy production locations or a refrigerated room. The use of automation results in improved precision because of reduced operator errors compared with manual methods of SPE. For these reasons automation provides for better utilization of resources.

There are many types of automation equipment for SPE. They include semi-automated instruments, workstations that carry out the entire SPE operation without intervention, and robotic systems that carry out many activities besides SPE and are specially customized for the user. Finally, there are on-line SPE–HPLC systems that allow the user to merely add the sample to the autosampler and analyse the sample directly. The concept of on-line SPE is that a sample is pumped and processed onto the SPE cartridge while the liquid chromatograph or gas chromatograph is processing the preceding sample.

See also: **II/Chromatography: Liquid:** Column Technology. **Extraction:** Solid-Phase Extraction. **III/Solid-Phase Extraction with Cartridges. Solid-Phase Extraction with Discs.**

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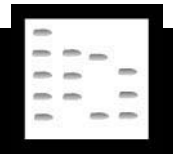
SPACE EXPLORATION: GAS CHROMATOGRAPHY



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The development in the past 40 years of space exploration has brought important information about the formation and evolution of the solar system and has opened a broad study of organic matter and its continuous chemical evolution which led to the appearance of life.