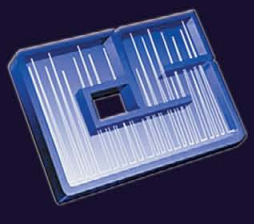
Chromatography Sample Preparation Handbook

Including Cost Saving Tips



CHROMATOGRAPHIC SPECIALTIES INC.

BRIEF INTRODUCTION — SAMPLE PREPARATION TECHNIQUES

Sample preparation plays a significant role in the quality of analytical results; it helps ensure that samples are in a suitable form to be easily detected using simple, robust analytical methods. Despite this, we often put our efforts into refining analytical methods rather than improving the methodologies of gathering and preparing the sample itself.

Sample preparation techniques clean, concentrate and if necessary, modify the analytes prior to analysis. Clean-up and concentration steps improve signal-to-noise ratios. Clean-up removes interfering material, thereby lowering noise levels; it also helps to preserve columns and instrumentation parts by protecting them from contamination. Concentration steps increase the signal and detectability by increasing the amount of analyte present in a given volume of sample.

This handbook provides an overview of the various sample preparation techniques available; our goal in creating this brochure was to help our customers answer two questions:

- **Can I improve my sample preparation methods for greater speed, cleanliness or concentration?**
- **What techniques are most likely to be beneficial to me?**

To do so, we've laid out this handbook as follows:

- The first 8 pages overview various sample preparation techniques commonly used in today's laboratories. They are grouped by sample matrix, going from liquid, through solids to gases. These pages highlight the pros and cons of each technique vis-à-vis the others and should help identify which techniques are most suitable for your lab.
- We follow those with 4 pages of industry specific examples of challenging analyses. On those pages, we explain the challenge then provide an overview of the method used to address the issues.
  - We have over 500 sample preparation applications available, spanning many different types of samples and techniques. We're confident we can help you improve your methods.
- The last half of the handbook introduces products that CSI has available to address your sample preparation needs.

We encourage you to consider the techniques used in your laboratory. Should you wish to discuss your current sample preparation processes or investigate potential improvements, please do not hesitate to contact us.

It is estimated that 60-80% of the work done in an analytical lab is spent on sample preparation. We can help you increase your productivity and reduce costs.

Analytical Method Improvements

- Take advantage of products that can pump out results in less time, while consuming less solvent and minimizing the need for retesting due to out-of-spec results
- Many sample preparation techniques can be automated to help save on labour costs and improve consistency

Technical Support and Instrumentation Assistance

Need expert advice? Our friendly and knowledgeable Technical Support and Instrumentation Teams consist of experienced chromatographers who understand the challenges facing separation scientists. They are available to assist you with everything from method reviews, to application support and troubleshooting.

Call Us

Our receptionist, order desk, customer service and technical support departments, in fact our entire company, is staffed by real people. Are you tired of trying to negotiate your way through frustrating automated voice systems? Call us! We'll be happy to help you.

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Wait! There's more…

We offer many more sample preparation products, as well as products for sample collection.
Proper sample collection also plays an important role in obtaining quality results.
Contact us for more information.

Life Science Products
Our sister company, MJS BioLynx, offers products for biological sample preparation.
Visit **www.biolynx.ca** or call **1-888-593-5969** for more information.

Throughout the handbook, look for these symbols, they'll provide information to illustrate how we can help you save time, money and effort…

Great ideas to help you and your lab reduce costs.

Industry specific examples of challenging analyses. Contact us for help with your specific application.

Tips and Tricks to help you get better results.

Free samples are available for certain products. Contact us and try these products in your lab.

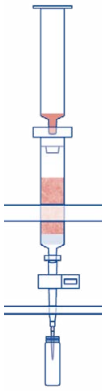
Save time so you can focus on other tasks or process more samples.

Contact our Technical and Instrumentation teams for more information or for assistance.

Think Green! Help the environment by using less solvent or generating less waste.

Solvent Extractions

Classic extraction methods where analytes are extracted into a liquid solvent, typically a high volatility non-polar organic solvent.

Technique	Advantages	Disadvantages	Notes	
Liquid-Liquid Extraction (LLE) **Sample Type:** Liquid **Purpose:** Removal of organic analytes from aqueous solutions	Very basic equipment Tolerates very dirty samples	Labour intensive Large amount of solvents used Solvent choices are limited to immiscible solvent pairs	Concentrates analytes (with addition of solvent evaporation step) The extraction is most often performed in a separation funnel Two immiscible solvents are shaken together, the analyte partitions into its preferred solvent Competing techniques: SPE, SLE, SPME	
Supported Liquid-Liquid Extraction (SLE) **Sample Type:** Liquid **Purpose:** Removal of organic analytes from aqueous solutions	Faster than LLE, eliminates emulsions, lowers solvent consumption Offers some advantages of SPE without requiring full method development	Less specific than SPE Volume of sample for extraction is very limited compared to either LLE or SPE	Modification of LLE Aqueous sample is adsorbed on diatomaceous earth, then extracted by passing immiscible organic solvent over sorbed sample Competing techniques: LLE, SPE	
Solid Phase Extraction (SPE) **Sample Type**: Solids and liquids **Purpose**: Removal of impurities and concentration of analytes	Compared to LLE: Saves time and money Uses less solvent Achieves better recovery and cleaner samples to better protect instrumentation Can be automated Broader applicability than SPME	Slower than SPME for SPME-appropriate samples	A sorbent is used to extract analytes of interest Solvents of different polarity are used to wash, condition and ultimately separate the analytes of interest from interfering compounds Competing techniques: LLE, SLE, SPME	 See page 16

Technical Support

Need expert advice? Our friendly and knowledgeable Technical Support and Instrumentation Teams consist of experienced chromatographers who understand the challenges facing separation scientists. They are available to assist you with everything from method reviews, to application support and troubleshooting. This is a free service for our customers; we will not charge for helping you resolve a chromatography problem.

Solvent Extractions (Cont'd)

Technique	Advantages	Disadvantages	Notes	
Dispersive SPE (dSPE) **Sample Type:** Solids and liquids **Purpose:** Removal of impurities	Faster than column SPE	Not as thorough as column SPE	SPE packing material is added directly to a solution rather than passing it through the packed material in a cartridge or tube The SPE sorbent is removed by centrifugation or filtration Competing techniques: column SPE	
QuEChERS **Sample Type:** Solids and liquids Suited for pesticide extracts in foods and similar products such as marijuana, wines, cooking oil, soils, etc. **Purpose:** Removal of impurities and concentration of analytes	Faster and lower solvent use than Soxhlet Uses lower toxicity solvents Works better with physically dirty samples than PLE	Less automated than PLE	QuEChERS stands for Quick, Easy, Cheap, Effective, Rugged, Safe Involves two steps: 1) Salting out extraction using buffered or unbuffered solvent (usually acetonitrile) 2) Dispersive SPE (dSPE) in which solid adsorbent is used to treat an aliquot from step 1 to remove interferences Ideal for selective detectors such as MS Competing techniques: Soxhlet, PLE	 See page 22
Soxhlet Extraction **Sample Type:** Solids **Purpose:** Removal and concentration of organic analytes (with addition of solvent evaporation step)	Only basic equipment required	Slow Requires large volumes of organic solvents Requires continuous flow of cooling water	Solvent is refluxed through the sample, which is held in a porous thimble Analytes of interest are collected and concentrated in an organic solvent Competing techniques: Pressurized Liquid Extraction (PLE), QuEChERS	 See page 25

TIPS

Choosing between dispersive and column SPE

Dispersive SPE is a fast technique to remove gross impurities from your sample. As the sorbents are simply mixed into a liquid sample then removed by centrifugation the technique is very fast and requires almost no operator attention, but it only has an adsorption step, limiting its selectivity. Column SPE adds washing and selective elution, so contaminants that are both more and less attracted to the solid phase than is the analyte are removed. However, it is slower and can require more operator attention.

Solvent Extractions (Cont'd)

Technique	Advantages	Disadvantages	Notes	
Pressurized Liquid Extraction (PLE) **Sample Type**: Solids **Purpose:** Removal and concentration of organic analytes	Fast Reproducible Automated	Requires large capital investment Pressure vessels need to be cleaned before reuse (not practical to use disposable vessels) Particle breakthrough can damage expensive equipment	Organic solvent is applied to the sample at high pressure and temperature (above normal boiling point) Competing techniques: Soxhlet, QuEChERS	

Concentration

Concentration is usually done after solvent extraction. The extracting solvent is evaporated off, leaving the less volatile analytes concentrated in a small volume of solvent. It can be taken to the extreme of evaporating all the original solvent and replacing it with a more polar (less volatile) solvent in solvent exchange.

Kuderna Danish **Sample Type:** Extracts **Purpose:** Concentration after extraction to liquid phase	Best recovery of volatile compound due to reflux action Can process 6-12 samples at a time	Time consuming	Start volume: 500 mL End volume: 0.5 mL	
Hybrid Evaporation **Sample Type:** Extract **Purpose:** Concentration after extraction to liquid phase	Handles large and small samples Good recovery even on volatile compounds Moderate speed Built in drying system Total automation	More expensive than using a single method	Start volume: up to 200 mL End volume: directly in vial down to 500 µL Combines vacuum, blowdown and Kuderna Danish	
Rotary Evaporator **Sample Type:** Extracts **Purpose:** Concentration after extraction to liquid phase	Handles large samples Concentrates individual samples quickly	Volatile components tend to have medium recovery Final volume often too high for analysis, requiring secondary evaporation Can process only one sample at a time	Start volume: up to 1L End volume: 5 to 10 mL (including rinses)	

Concentration (Cont'd)

Technique	Advantages	Disadvantages	Notes	
Nitrogen Blowdown **Sample Type:** Extracts **Purpose:** Concentration after extraction to liquid phase	Can process up to 60 samples at a time Can accommodate a variety of sample sizes	Low recovery of volatile compounds Higher running costs due to gas consumption Usually all samples must be in the same size tube Evaporation slows down later in run unless pressure is raised	Start volume: 1 to 200 mL End volume: 0.2 to 0.5 mL	 See page 21
Dialysis **Sample Type:** Liquids, Proteins **Purpose:** Contamination removal and desalting	Simple and easy to set-up Gentle Low cost	Slow Lack of specificity	Exchange of buffer medium across a semi-permeable membrane New dialysis tubes incorporate dialysis membrane in cap to reduce sample loss	

Extraction to Sorbent Phase

Extractions to sorbent phases depend on partitioning between a liquid or gas phase and solid or bonded "pseudo-liquid" phase to separate and concentrate analytes. Following adsorption (to a solid) or absorption (in a pseudo-liquid), either the surrounding solvent or the temperature is changed to drive the analytes into the final phase for analysis.

Solid Phase Microextraction (SPME) **Sample Type:** Liquids, gases and headspace over solids or liquids **Purpose:** Concentration of analytes	Fast and clean	Largely limited to volatile or semi-volatile compounds	Fused silica fiber is coated with polymeric stationary phase Analytes diffuse and partition / adsorb onto stationary phase Analytes are then thermally desorbed by placing fiber into GC injection port Competing techniques: PLE, LLE, SPE	 See page 31

Instrumentation Support

Our Instrumentation Team is available to answer your questions and perform calibrations and repairs to your instrumentation. They can also customize instruments, and perform difficult modifications and installations to fit your specific applications or needs.

Extraction to Sorbent Phase (cont'd)

Technique	Advantages	Disadvantages	Notes	
Thermal Desorption **Sample Type:** Volatiles in solids, liquids or in gas phase **Purpose:** Extraction and concentration of volatile organic compounds (VOCs)	Allows for trace level analysis No solvents required Extraction and concentration in one step Little to no sample handling Clean GC injection	Strictly for volatiles Requires specific instrumentation	Samples are pumped through a sorbent material (Trap). Trapped volatiles are then desorbed to GC or GC/MS. With special equipment, near real-time monitoring can be performed Competing techniques: Bulk sampling bags or sample canisters	 See page 26
Purge and Trap - Dynamic Headspace **Sample Type:** Volatiles in liquid or solid samples **Purpose:** For extracting and concentrating volatile organic compounds (VOCs) from liquids and solids	Allows for trace level analysis No solvents required Extraction and concentration in one step Little to no sample handling Cleaner GC injection	Strictly for volatiles Limited sample size (for purge and trap) Requires specific instrumentation	Samples are purged with inert gas to drive volatiles to a sorbent material (Trap). Trapped volatiles are then desorbed to GC or GC/MS. With special equipment, Dynamic Headspace can be performed on large objects Competing techniques: Solid Phase Micro Extraction, Static Headspace	 See page 26

Static Headspace

When analytes are volatile and the solvent is (relatively) non-volatile, heating a closed vessel concentrates the analytes in the gas phase above a liquid or solid sample. Sampling this head-space with a gas-tight syringe allows a simple transfer of the desired volatile compounds to a GC, while eliminating any relatively non-volatile compounds which would otherwise accumulate in the GC inlet and the head of the GC column.

Static Headspace **Sample Type:** Volatiles in liquid, solid or mixed matrices Pharma, food, and biological **Purpose:** Extraction of VOCs from complex matrices	No solvents required Extraction and concentration in one step Little to no sample handling Cleaner GC injection	Strictly for volatiles Limited sample size Typically requires specific instrumentation	Volatiles are forced from the matrix to attain an equilibrium between phases Heat, agitation and time are used to drive the equilibrium Competing techniques: Solid Phase Micro Extraction, Purge & Trap	 See page 27

Supercritical Fluid Extraction (SFE)

A modern revision of solvent extraction, SFE replaces organic solvents with supercritical fluids. As they are neither gasses nor liquids, the viscosity and density of supercritical fluids can be controlled over significant ranges to allow better, faster extractions.

Technique	Advantages	Disadvantages	Notes	
Supercritical Fluid Extraction (SFE) **Sample Type**: Bulk plant materials **Purpose:** Extraction from solids	Uses inexpensive, non-toxic solvents Fast Automatable	High cost of equipment Limited selectivity	Sample is placed in a flow-through column and supercritical fluid (typically CO_2) is passed through the sample. The density and viscosity of CO_2 can be controlled. The extracted analyte is collected in solvent or trapped on adsorbent. Collecting pure analyte is simple as CO_2 returns to gas under ambient conditions Automated and manual versions available Competing techniques: Quechers, Soxhlet, PLC	

Particle Removal

Solid particles in a liquid sample can plug and erode sensitive instruments. These techniques perform simple physical separations to eliminate particles of all types.

Filtration **Sample Type**: Liquid **Purpose:** Particle removal or concentration of particles as a solid sample	Cleaner product than centrifugation Handles larger volume samples than centrifugation or syringe filtration Helps extend column and instrument life No effect on dissolved chemical contaminants	Handles fewer samples at a time than centrifugation Doesn't handle small volume samples well	Some filters work with gravity flow Finer filters require support and vacuum for reasonable speed Competing techniques: Syringe filtration, centrifugation	
Syringe Filtration **Sample Type**: Liquid **Purpose:** Particle removal	Cleaner product than centrifugation Better than standard filtration for small samples	Handles fewer samples at a time than centrifugation Doesn't handle large volume samples well	Attaches to luer or luer lock syringe Syringe provides pressure for faster filtration Competing techniques: filtration, centrifugation, spin filtration	 See page 28

Particle Removal (Cont'd)

Technique	Advantages	Disadvantages	Notes	
Spin Filtration **Sample Type:** Liquid **Purpose:** Remove particles from liquid samples	Cleaner product than pure centrifugation Better than standard filtration for small samples Handles batches of samples	Doesn't handle large volume samples well Relatively expensive due to cost of centrifuge	Centrifugal force is used to press liquid through membrane filter	 See page 23
Centrifugation **Sample Type:** Liquid **Purpose:** Separation of substances with different densities	If the tubes are reused, the process may be less expensive Less interaction between sample and various filter membranes	Can be a slow process Relatively expensive due to cost of centrifuge	Uses centrifugal force to separate 2 or more components in a mixture or suspension Samples are placed in specialty tubes (disposable or reusable) and spun at high speeds to create a separation Competing techniques: Syringe, membrane and paper filtration	

Derivatization

Derivatization chemically modifies analytes to improve volatility for GC separations or to increase their UV absorption or fluorescence for LC detection. Both can make analytes more visible in an analysis without increasing concentrations.

Derivatization **Sample Type:** Liquid or solid **Purpose:** Alteration of difficult-to-analyze samples to make them more visible to GC or LC detectors	Provides increased sample volatility Improved selectivity Enhanced detectability	Reagents are often moisture or air sensitive Residues build up in GC detectors Reagents often harmful to GC column phases	Generally, three methods of derivatization: Silylation, Acylation and Alkylation Competing techniques: in some cases, pyrolysis + FT-IR or another spectroscopy method	 See page 30

Our Technical Team is only a call away!

Contact us for more information on Sample Preparation Techniques.

Precipitation

Precipitation changes the chemical conditions in a sample to cause undesired solutes to precipitate out, for easy removal by filtration or centrifugation. It is often used to remove proteins, but can also work for salts.

Technique	Advantages	Disadvantages	Notes	
Protein Precipitation **Sample Type:** Serum, plasma, other bodily fluids **Purpose:** Bulk removal of proteins	Quick, inexpensive protein removal	Precipitated proteins can trap analytes Addition of organic solvent can make injection solvent too strong in RP HPLC	Adding polar organic solvent (acetonitrile, methanol) to sample denatures proteins, causing precipitation Precipitate is removed by filtration Also known as protein crash	
Immunoprecipitation **Sample Type:** Cell or tissue lysates **Purpose:** Enrichment of specific proteins	Highly specific for desired proteins	Elution can sometimes result in low protein recovery and antibody contamination	Can be used to remove high abundance protein in order to analyze low abundance proteins	

Affinity Chromatography

Affinity chromatography involves the removal (or in some cases concentration) of analytes by antibody – antigen interactions. This technique originally, and still largely, uses monoclonal or polyclonal antibodies; however, some analytes can now be targeted by specially synthesised polymers with tailored molecular level pockets.

Affinity Chromatography **Sample Type:** Bodily fluids, cell or tissue lysates **Purpose:** Enrichment of specific proteins	Highly specific High recovery for small volume samples	Elution can sometimes result in low protein recovery and antibody contamination Requires column derivatized with suitable antibodies	Can be used to remove high abundance protein in order to analyze low abundance proteins	

Sample Prep Automation

Process more samples in less time

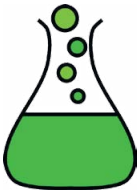
- ⇨ **Minimize human error and increase reproducibility**
- ⇨ **Reduce hands-on time so you can focus on other tasks**

Despite the initial cost of the instrumentation and the labour investment needed to integrate the system into the workflow, the benefits (increased turn around time, high sample throughput, better consistency and decreased need for re-analysis) lead to a very good return on investment. In most cases, the payback period is between 9-18 months, allowing your company to lower the cost of each analysis for extra profit or greater competitiveness.

Contact us for a **FREE Consultation**

Fast, proven, sample clean-up for pesticides and cannabinoids in marijuana edibles.

Marijuana edibles are particularly difficult samples to analyse, both for forensic labs assessing seized products and for licensed medical marijuana producers. Fats and sugars included in edibles are notorious for contaminating instruments and columns.



Application Example

Contact us for help with your specific application.

The techniques outlined here can be applied to other foods or to active compounds in Natural Health Products.

Procedure: (Abbreviated method. Contact us for full details.)

1. **Sample Homogenization:**
   (similar processing is used for all extraction techniques)
   - Hard Candies and Chocolates - ground to a fine powder using a freezer mill.
   - Gummy Candies - cut into slim pieces.
   - Carbonated Beverages - degas for 30 min by sonication.

2. **QuEChERS Extraction**
   <2 min / sample and <90 min / batch, <15 min / batch for carbonated beverages.
   a. Carbonated beverages: To a 50mL centrifuge tube add 10mL of the degassed sample and internal standard.
      Candies, baked goods, or oil: To a 50mL centrifuge tube add 1g of the pre-treated samples, internal standard and 10mL of reagent water. Hydrate for 1 hour using a horizontal shaker.
   b. Add 10mL of acetonitrile (MeCN) with 1% acetic acid.
   c. Add QuEChERS extraction salts from pouches (Product # UCTECQUUS950CTMP), and vortex for 10 seconds.
   d. Shake vigorously for 1 minute. For gummy samples, add 2 metal balls and shake for 10 minutes.
   e. Centrifuge for 5 minutes.

3. **Dispersive SPE Clean-up for Pesticide Residue Analysis**
   <2 min / sample and <10 min / batch
   a. Transfer 1mL of the supernatant to 2mL centrifuge tube pre-loaded with dSPE sorbents Product # UCTECQUUS142CT)
   b. Shake vigorously for 1 minute; centrifuge for 5 minutes.
   c. Vortex 200μL extract with 200μL of DI water in a 2mL vial for 30 seconds; inject.

4. **Serial Dilutions for Cannabinoid Analysis**
   a. Dilute QuEChERS extracts to 100 to 200 ng/mL of cannabinoids based on expected concentrations.
   b. Spike the diluted samples with target cannabinoids; quantify using the standard addition method.

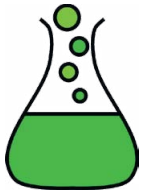
Ready for analysis of pesticide residues and potency in approximately 2 hours.

QuEChERS is:

- An effective method for fast reduction of multiple contaminant classes from samples.
- A “just-enough” technique: capture analytes as soon as just enough clean-up is done for simple analysis.
- A broadly useable technique for many classes of analytes.
- Applicable to many formats of natural health products or functional foods. Assess safety and efficacy with one clean-up.

Copolymeric SPE Column Extraction of Barbiturates from Body Fluids or Tissue

Bodily fluids and tissues are challenging matrices to analyze. Proteins, fats, sugars and salts will all be present to some degree, and can all interfere with GC or HPLC analysis.



Application Example

Contact us for help with your specific application.

Procedure: (Abbreviated method. Contact us for full details.)

1. **Prepare for Extraction:**
   <2 min sample of handling, 15 minutes / batch non operator time
   a. Mix 1 mL of 100 mM phosphate buffer (pH 6.0), internal standards and 1-2 mL of blood, plasma/serum, urine, or 1g homogenised tissue. Mix vigourously and let stand for 5 minutes.
   b. Add 2 mL of 100 mM phosphate buffer (pH 6.0). Mix vigourously.
   c. Sample pH should be 6.0 ± 0.5, adjust pH sodium phosphate if needed.
   d. Centrifuge for 10 minutes at 2000 rpm and discard pellet.

2. **Condition CLEAN SCREEN® Extraction Column:**
   Approximately 2 minutes / 24 column batch
   (Product # UCTZSDAU020. The stepped tube design concentrates more sample on less stationary phase.)
   a. 1 x 3 mL CH_3OH
   b. 1 x 3 mL D.I. H_2O
   c. 1 x 3 mL 100 mM phosphate buffer (pH 6.0)
   NOTE: Aspirate at full vacuum or pressure.

3. **Apply Sample to Column:**
   1.5-3 minutes per batch of 24 columns
   a. Pour supernatant from step 1 on top of freshly conditioned column.
   b. Aspirate through column at 1 to 2 mL/minute.

TIPS

Save time on sample prep – condition your columns while centrifuging your tissue samples!

4. **Wash Column:**
   10 minutes per batch of 24 columns
   a. 1 x 3 mL D.I. H_2O
   b. 1 x 1 mL 100 mM Acetic Acid
   c. Dry column (5 minutes at full vacuum or pressure).
   d. 1 x 2 mL hexane

TIPS

Drying columns between steps allows immiscible solvents to be used in successive steps. If miscible solvents are used, no drying is needed. This is an advantage over LLE: you can use miscible solvents without risk of non-specific extractions.

5. **Elute Barbiturates:**
   1.5-3 minutes per batch of 24 columns
   a. 1 x 3 mL Ethyl Acetate:Hexane (50:50)
   b. Collect eluate at 1 to 2 mL/minute.

6. **Dry Eluate:**
   Time depends on available dry down tools
   a. Evaporate to dryness at < 40°C.

7. **Reconstitute / Derivatize:**
   2 minutes / batch
   a. LC-MS/MS: Reconstitute sample in 100 µL of mobile phase; inject 10 µL.
   b. GC-MS: Dissolve residue in 100 µL of Ethyl Acetate.
   Optional Derivatization for Increased Volatility.
   Alkylate with TMPAH (Reaction occurs in GC injection port! No added time).

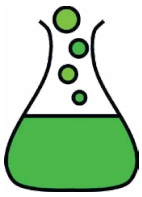
Advantages of CLEAN SCREEN® DAU and other Copolymeric SPE Phases:

- Thorough removal of multiple contaminant classes such as protein, fats, and sugars through high specificity and broad retention. Reversed phase and ion exchange mechanisms, two ways to retain analytes or contamination. Visit **www.chromspec.com/spe-app** for examples of different extractions.
- Excellent loading and retention. Both phases are on every particle, minimal analyte movement when changing retention mechanisms.

Clean-Screen DAU is optimized for general drugs of abuse. Contact us for other special or general purpose copolymeric phases.

Column SPE/QuEChERS Procedure for Analysis of Bisphenol A in Canned Food Products

Humans put the "omni" in omnivorous, our foods may be animal or vegetable, fatty or lean, full of pigments or nearly white. This makes extraction and clean up from foods a particular challenge, but QuEChERS can handle all of these conditions.



Application Example

Contact us for help with your specific application.

Procedure: (Abbreviated method. Contact us for full details.)

1. **QuEChERS Extraction**
   <2 minutes / sample and 5 minutes / batch for centrifugation
   a) Add weighed sample and internal standard to a 50mL QuEChERS tube
   b) Add 10mL of acetonitrile and mix
   c) Salt out acetonitrile from water with QuEChERS salt mix (Product # UCTECQUEU750CTMP)
   d) Shake vigorously for 2 min. and centrifuge for 3 min.

2.1. Column SPE Clean-up for Lightly Pigmented Food
<10 minutes – PSA and C18 for lipid removal
a) Attach Product # UCTECPSAC1856 to the vacuum manifold and add Na_2SO_4 (Product # UCTECSS10K)
b) Condition with 2 x 2mL of acetonitrile
c) Load the supernatant and collect the extract
d) Concentrate to dryness

2.2 Column SPE Clean-up for Highly Pigmented Food
<10 minutes – PSA and GCB for lipid and pigment removal
a) Attach Product # UCTECPSACB6 to the vacuum manifold and follow the same steps as 2.1

3. Derivatization and Analysis
a) Reconstitute and silylate; analyze by GC/MS.
For information on derivatization, including silylation, reagents and their uses, please see page 30 or visit **www.chromspec.com/derivatization**

With column SPE, different phases can be run in series or different clean-up steps can be run simultaneously by using column adapters to stack columns on top of each other as they go through a vacuum manifold.

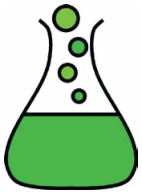
QuEChERS is a flexible technique

QuEChERS normally uses dSPE for greater speed. However, with challenging matrices such as prepared foods with multiple ingredients, column SPE can be used for more effective clean-up. This flexibility has allowed QuEChERS to evolve from a technique for fruits and vegetables, meats and seafood, to include soil samples and even products such as baby toys.

Visit **www.chromspec.com/quechers** for more information.

Extraction and Sample Concentration from Large Volumes of Water

Biomagnification in the food chain makes detecting low concentrations of organic contaminants in water crucial.



Application Example

Contact us for help with your specific application.

This analysis concentrates pesticides 500:1 from water samples.

Procedure: (Abbreviated method. Contact us for full details.)

EPA Method 8141B - Determination of Organophosphorus Pesticides and Triazine Herbicides in Water by Solid Phase Extraction.

1. **Sample Spiking**
   a. Add surrogate standard to 1L portions of neutral water samples (Note: use of smaller sample volumes is permitted if method sensitivity is not an issue).

2. **SPE Cartridge Conditioning**
   a. Wash the SPE cartridges with 3 x 5mL DCM
   b. Condition cartridges with 2 x 5mL methanol.
   c. Equilibrate the cartridges with 2 x 5mL DI water.

3. **Sample Loading**
   a. Connect water reservoirs to SPE cartridges with transfer lines. Draw samples through in a fast drop-wise fashion (10-15 mL/minute).
   b. Remove the transfer lines, dry cartridges under vacuum for 10 minutes.

4. **Eluate Drying and Elution**
   a. Attach the drying cartridges below SPE cartridges, place collection vials in manifold.
   b. Rinse bottle, transfer tube, elute SPE cartridge with 5mL acetone, 10mL DCM.
   c. Remove the transfer tubes, elute SPE cartridges with 5mL DCM.

5. **Eluate Concentration**
   a. Concentrate the eluates to about 0.5mL under a gentle stream of nitrogen at 40°C.
   b. Rinse walls of vials with 3mL of n-hexane, concentrate to about 2mL.
   c. Transfer the extracts to 2mL vials, adjust the volume to 2mL with n-hexane.
   d. Add internal standard, samples are ready for GC analysis.

TIPS

1/8" OD PTFE tubing and an appropriate column adapter allow you to extract water samples ranging in volumes from hundreds of millilitres to liters straight from the sampling bottle.

Column SPE and transfer lines provide you with:

- Simple, reliable extraction of large volumes of water with minimal operator time
- A 50:1 concentration step occurs **before** sample blowdown!
  (Compared to typical 5:1 concentrations with LLE)
  - For some compounds, extraction can provide an even greater concentration ratio.
  - Final 10:1 concentration is from just 20mL of solvent.
- Flexibility to use water miscible solvents to extract polar organics from water samples.

Visit **www.chromspec.com/spe-app** for more information.

SPE is an extremely powerful and flexible way to concentrate and purify analytes of interest and remove contaminants from sample matrices. SPE products have been developed for specific applications using a wide range of sorbents, formats and sizes. Here, we provide a brief introduction to selecting from these different SPE products.

For more information, please contact our Technical team.

Using SPE instead of LLE can save you time and money

- Faster processing with fewer steps and less sample handling
- Uses less solvent and glassware
- Does not require the use of immiscible solvents, simplifying solvent selection
- Greater analyte recoveries, so you can work with smaller samples
- Lower risk of cross contamination
- Highly reproducible extractions for better results
- More specific extracts for better equipment protection
- Creates less disposable waste, which helps the environment

SPE Formats

SPE Columns

⇨ A flexible, reliable, cost effective solution for laboratory extractions

SPE columns are ideal for laboratory extractions when a vacuum or positive pressure manifold or SPE autosampler is available. Their minimalist design cuts cost and waste, and allows for easy packing to suit all needs.

SPE columns are made of polypropylene or glass, are available in a variety of shapes and have volumes of approximately 1mL to 50mL. Columns may be attached in series or with large volume reservoirs, using adapter fittings. A certain weight of sorbent material is contained in the column between an upper and lower frit. They are primarily used with vacuum or positive pressure manifolds to move samples through the column. Automated systems are available (see page 20).

The UCT SPE Column line includes products specifically designed for use in:

- **Clinical and Forensic** – for the extraction of acids, neutrals and bases; ideal for both screening and confirmation analyses of virtually all drug categories
- **Environmental** – wide range of cartridge sizes, sorbent types tailored to the specific requirements of each analysis
- **Toxicology** – extract a wide array of basic drugs including benzodiazepines, opiates and THC metabolite
- **General Sample Clean-up** – of polar, non-polar and ionic compounds

Push-Thru Cartridges

⇨ Excellent for field sampling and avoiding carryover

With luer connectors at the top and bottom of each unit, Push-Thru cartridges allow samples to be loaded, washed, or eluted using a simple luer tip syringe. Just slip the syringe into the female luer inlet of the cartridge and push sample through with the syringe.

You can choose from a wide range of sorbent types tailored to the specific requirements of each analysis. Excellent for field sampling, and avoiding carryover. Can be used with vacuum manifolds if needed.

The UCT Push-Thru Cartridge line includes products specifically designed for use in:

- **Environmental** – Capture and concentrate field samples with light weight and durable equipment. Transporting sample sorbed to SPE phase eliminates weight, chance of spills, and can provide additional stability. UCT's specialty Glyphosate Push-Thru cartridges strip common matrix components interfering with glyphosate ionisation and detection in LC/MS.
- **Clinical and Forensic** – Push-Thru cartridges combined with standard disposable syringes provide a simple inexpensive single use flow-path at the sample collection point.
- **General Sample Clean-up** – Avoid tying up vacuum manifolds or automated extractors for method development and proof of concept for SPE. Test methods on single samples at the bench with a syringe and cartridge.

Extraction Disks

⇨ An ideal solution for large volume samples

The disk format provides a large surface area for sorbent/sample contact. Faster flow rates and higher throughput are realized compared to liquid-liquid extraction or traditional packed column technology. The particle-loaded membranes, or disks, are produced using the most common sorbent particles. They provide a more uniform extraction medium than can be achieved in a traditional SPE column or cartridge prepared with the same size particles. Extraction accessories include a manifold with reservoir, disk support and suitable collection vials. Automated systems are available (see page 21).

The 3M Empore™ and Horizon disk lines feature products specifically designed for use in:

- **Environmental** – For fast extractions of large water volumes. Common analytes of interest are persistent organic pollutants in surface waters or traces of oil and grease in waste water (EPA 1164).

Products include:

- Reverse phase
- Ion exchange
- Oil and Grease
- Metal Scavengers
- Activated Carbon
- Pre-filters for particle laden samples

TIPS

Symptoms of a Sample in Need of Further Clean-up:

- Poor reproducibility
- Column failure (high back pressure, changing retention times)
- Incorrect quantitation on QC samples

Fixing Insufficiently Clean Samples:

- Try a different sorbent which uses the same retention mechanism (i.e. try end capped silica C18 instead of non-end capped C18)
- Change to a different retention mechanism sorbent
- Change the wash solvent
- Change the ionic strength (when using ion exchange sorbents)
- Change the pH of the load and wash
- Change the % organic of the load and wash
- Contact our Technical Support Team to discuss your application and current method

SPE Sorbents

- SPE supports are mostly based on porous silica or polystyrene-divinylbenzene (PSDVB) particles, with or without derivatization
- Particles are typically 45-60 μm in diameter, but may be smaller or larger for specific methods
- Particles are typically irregular, but may be spherical for certain high performance applications
  - Large, irregular particles are very effective in SPE. Retention is binary (100% retained, or 100% eluted), so there is little need for high plate counts
- Materials other than silica or PSDVB (e.g. Florisil or graphitized carbon) are also used, mostly without derivatization

Hydrophobic (Reverse Phase) Extraction Sorbents

- Sorbent is composed of non-polar molecules bonded to support particles. PSDVB may be used underivatized
- Used to extract non-polar / neutral compounds out of complex matrices
- C18 phase is the most widely used because highly predictable hydrophobic retention
- Analytes are eluted with relatively non-polar solvent systems
- Common phases: C18, C8, Styrene-Divinyl Benzene (SDVB)

Hydrophilic (Normal Phase) Extraction Sorbents

- Sorbent is composed of polar organic molecules bonded to support particles. Silica, fluorisil and other inorganic particles may be used underivatized
- Compounds are retained through polar interactions e.g. hydrogen bonding, pi-pi or dipole-dipole.
- Analytes are eluted with relatively polar, sometimes aqueous solvent systems
- Common phases: Silica, Fluorisil, Cyano

Carbon, Graphitized Non-porous Sorbents

- **Carbon can isolate extremely polar organic compounds**
- Carbon adsorption combines hydrophobic mechanisms, high surface area and ion exchange
- This interaction can happen in a wide range of polar and non-polar solvents

Ion Exchange Sorbents

- Sorbent must carry opposite charge to analyte.
- Sorbent and the analyte must be ionized for retention
- Sorbent or compound must be neutral for elution
- Sorbent is composed of organic acids or bases bonded to particles
  - Anion Exchangers include Aminopropyl, Quaternary amine and Polyamine
  - Cation Exchangers include Carboxylic Acid, Benzenesulfonic Acid and Triacetic acid

When pH is within 2 units of pK_a acids and bases are in a mix of ionized and neutral form leading to irregular retention / elution.

	Cation	Anion
$pH < pK_a-2$	+	0
$pH > pK_a+2$	0	-

Sorbent Selection Considerations

- Does the analyte appear to be polar or non polar?
- Are the analytes soluble in the matrix (and eluent)?
- Do the analytes contain any ionic groups?
- What is the concentration of the analyte in the sample?
- Are the compounds stable in acid or base?
- Are you derivatizing for GC or HPLC analysis?

The answers to these questions will help determine which clean-up techniques or products might work for your particular application.

Contact our Technical Support Team for further discussion.

Mixed Sorbents

- Mixed chemistry is beneficial when looking to extract both a neutral and a charged compound or amphoteric molecules from polar and non-polar molecules
- Copolymeric mixed sorbents are composed of types of multiple functional chains bonded to the particle. One ion exchanger or polar chain, one hydrophobic carbon chain
  - Produced with equal parts of each functional group bonded to the particle
  - Yields reproducible bonded phases and allows the maximum use of mixed mode separation mechanisms
- Mixed bed systems have one phase on any given particle, mix the particles for dual retention modes

Covalent Extraction Sorbents

- Ideal for scavenging reactive compounds in synthetic chemistry
- Covalent sorbents have either epoxy, aldehyde, isocyanate or thiopropyl functional groups that are bound to the silica backbone by a hydrocarbon chain
- These groups react selectively with analyte functional groups creating a permanent chemical bond between the stationary support and the analyte

Visit **www.chromspec.com/spe** for more information.

We offer SPE products from these trusted suppliers:

3M Empore™

Horizon technology
PREPARE FOR BETTER RESULTS

UCT PARTNER

MN MACHEREY-NAGEL

RESTEK Pure Chromatography

TRY IT NOW!

FREE SAMPLES are available!

Free Catalogue!

Get the UCT Solid Phase Extraction Products catalogue.
Request Literature Code **UCT27**

Why choose an automated system?

- Process more samples in less time
- Minimize human error and increase reproducibility
- Reduce hands-on time so you can focus on other tasks

Contact us for a FREE consultation and a Return on Investment calculation.

FREE Consultation

LCTech FREESTYLE™

⇨ Mycotoxins and Small Samples

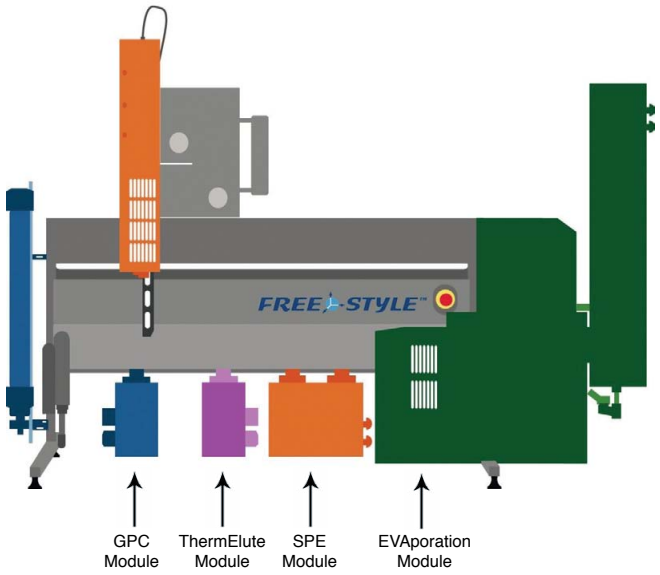
Flexible, Precise, Modular

The FREESTYLE™ is a fully automated sample preparation system for the unsupervised processing of food, animal feed, and pharmaceutical samples.

The FREESTYLE™ BASIC base unit can be expanded with some or all of the following modules:

- **SPE Module** – for Solid Phase Extraction using columns between 1mL and 15mL
- **EVAporation Module** – for evaporation or solvent exchange
- **GPC Module** – for Gel Permeation Chromatography of samples with complex matrices
- **ThermELUTE Module** – for mycotoxin samples, this unique module can be integrated with any HPLC for a 100-fold increase in sensitivity

When several modules are installed, each unit can be utilized individually or in combination.



LCTech DEXtech™

⇨ Dioxin Samples

Designed for sample preparation for dioxin analyses, this system can be used for food, animal feed, and environmental samples.

- Fast processing: purify your sample in 50 to 60 minutes
- Low solvent consumption: 300 to 400mL per sample

Developed in collaboration with a leading laboratory for dioxin analysis in Germany, the CVUA Münster (Prof. Fürst / Dr. Bernsmann).

Horizon SPE-DEX® 4790

⇨ For large volume or dirty samples

Automated Solid Phase Extraction

- Programmable and multipurpose – works on a wide range of applications and sample types
- Uses 47mm disks, 90mm disks, or standard cartridges
- Processes samples directly from their original container
- Automatically rinses the sample container
- Preconditions the sorbent material within the SPE disk, passes the water sample through the disk, and elutes the sorbed analytes from the disk into a collection vessel

Horizon SmartPrep® Extractor

⇨ For small volume or clean samples

Total Walk-away Automation

- Positive pressure controlled via 6mL syringe
- Modular and scalable—up to 8 modules from one PC
- Process up to 12 samples sequentially
- Collect up to 4 fractions per sample
- Works with most cartridge sizes (1, 3, or 6mL)
- Independent methods can be applied to each sample

Horizon technology

PREPARE FOR BETTER RESULTS

Horizon XcelVap®

Automated Concentration

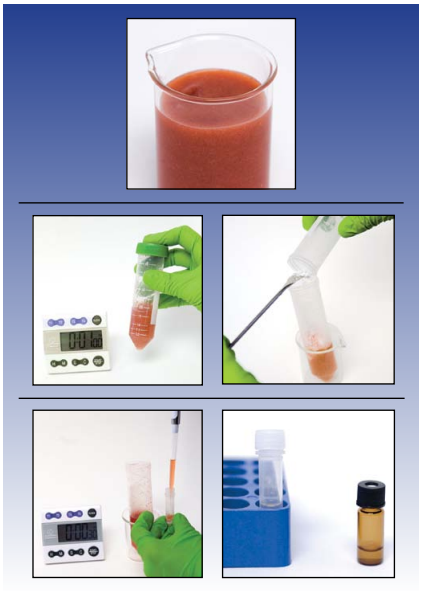
The XcelVap® Automated Evaporation System is a modern, compact benchtop laboratory concentrator that provides rapid, gentle evaporation of up to 54 sample extracts ranging in size up to 200mL each. Evaporation is accomplished by combining consistent heat, controlled sparge gas, and active venting of the solvent vapors.

- Automated pressure profiling for optimum speed with the most reproducible recoveries
- Nozzles can be individually changed by the user, simplifying maintenance and reducing operating costs
- Interchangeable racks accommodate practically any glassware on the market
- Easy water bath management with water level sensor and front draining
- Small footprint, portrait orientation and exhaust venting help to avoid the need for placement in a fume hood

Visit **www.chromspec.com/evaporation** for more information.

QuEChERS

⇨ Quick, Easy, Cheap, Effective, Rugged, and Safe clean-up method for pesticides and pharmaceuticals in food products, biological samples and similar analytes and matrices



QuEChERS are easy to use!
Prepare samples for LC or GC analysis in 3 simple steps:

Blend
- Homogenize the sample. Add water if required to give a smoothie-like consistency

Extract and Dry
- Transfer to a centrifuge tube, add acetonitrile and internal standard, then shake vigorously for 1 minute
- Add buffering salts and shake again. The salts trigger a phase separation between the water and acetonitrile extract. Centrifuge for 5 minutes to complete the separation of phases

Clean Up
- Transfer acetonitrile extract (supernatant) to a 2nd centrifuge tube, this one containing dSPE material
- Shake, centrifuge, and transfer supernatant to an autosampler vial, for analysis by GC or HPLC
- Note: Non-target organics are trapped with the spent dSPE material (which will be discarded)

UCT QuEChERS

Pre-packed products — save valuable time for increased lab throughput

- An extensive selection of QuEChERS extraction salts pre-packed in polypropylene centrifuge tubes or Mylar pouches
- Custom products are available

UCT PARTNER

Restek QuEChERS

Reusable QuEChERS Centrifuge Tubes

Cut costs, storage space and lab waste by eliminating one-time use PP tubes from your method

These sturdy, easy-to-clean FEP tubes last indefinitely under normal usage conditions.

QuEChERS Optimized Centrifuge
- Rugged, push button design, at a great price
- Meets or exceeds requirements of original unbuffered, AOAC, and European QuEChERS methodology

RESTEK
Pure Chromatography

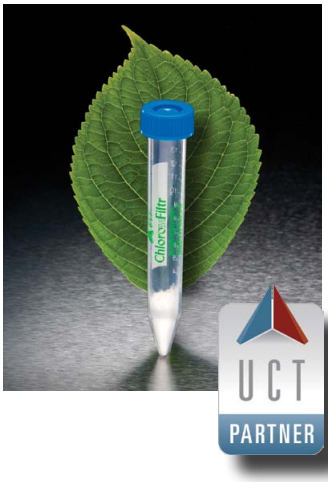
EXPERT ADVICE

We offer buffer salt packages designed to stabilise acid or base sensitive analytes, and dSPE sorbent packages to clean up extracts with different levels of fats, sugars or pigments. Contact our Technical Team for advice on matching kits to your samples.

Visit **www.chromspec.com/quechers** for more information.

UCT ChloroFiltr®

⇨ Selectively Remove Chlorophyll from Plant Extracts and Keep Planar Pesticides Intact



Standard QuEChERS products use Graphitized Carbon Black (GCB) to remove chlorophyll from samples; although GCB is effective, it also removes planar (polar aromatic) pesticides. Low recoveries for these analytes reduce analytical sensitivity and reproducibility. High polarity solvents can be used to remove the planar pesticides from GCB but cause an increase in background interferences (and an extra step). UCT uses ChloroFiltr® in place of GCB.

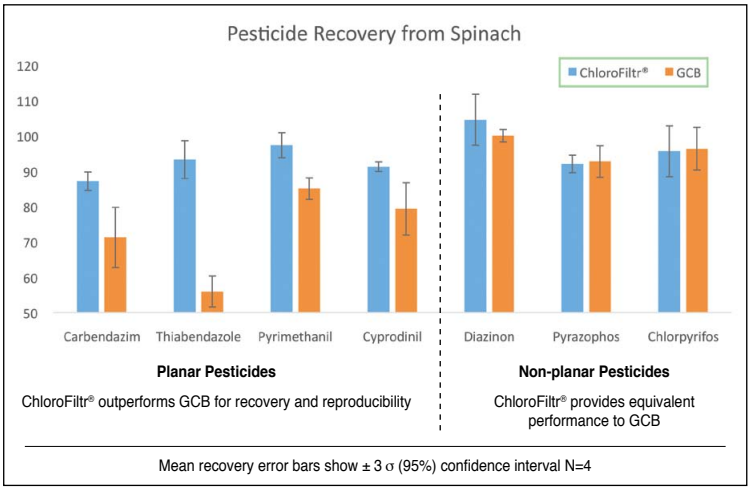
ChloroFiltr® has been tested against hundreds of pesticides and herbicides and has been shown to reduce chlorophyll concentration by greater than 82% without loss of planar analytes.

No method modification is necessary when substituting ChloroFiltr® for standard GCB-based materials.

ChloroFiltr® is available in a variety of formats to clean between 1 and 12 mL of supernatant.

Crude Spinach Extract | dSPE cleanup with ChloroFiltr® | dSPE cleanup with GCB

ChloroFiltr® has removed as much chlorophyll as GCB and pesticide recoveries are higher.



UCT SpinFiltr™

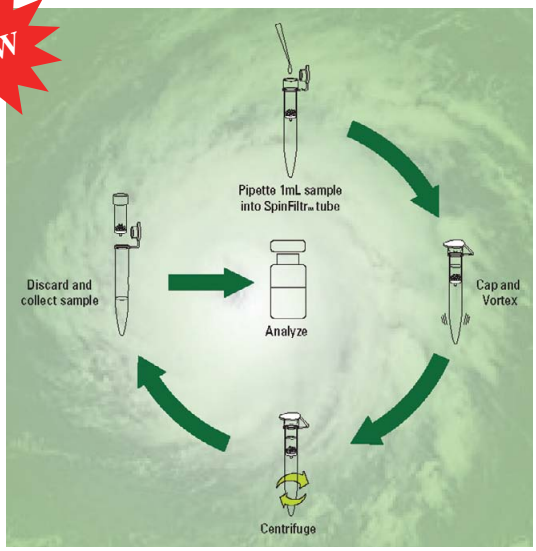
⇨ SpinFiltr™ makes dSPE even easier

⇨ 0.22µm filter ensures complete separation of sorbent from sample after centrifugation

SpinFiltr™ steps are the same as any dSPE:
1. Add sample to sorbent in upper chamber
2. Vortex/Shake
3. Spin – liquid flow through membrane

SpinFiltr™ A Whole New Spin on dSPE

NEW



Sorbent, drying salt and unwanted matrix stay in the top chamber while purified extract is filtered and collected in the bottom portion of the tube. The chamber is discarded and analysts are left with only purified extract that is ready for analysis.

In conventional dSPE, the liquid sample directly overlays a "pellet" of sorbent after spin down. Abrupt handling can easily re-suspend the sorbent and contaminate samples.

TRY IT NOW!

FREE SAMPLES are available!

Visit **www.chromspec.com/quechers** for more information.

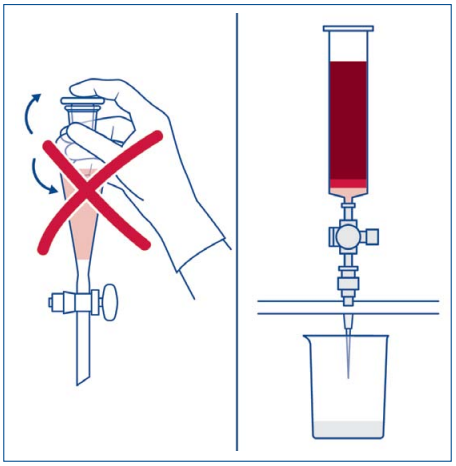
Macherey-Nagel CHROMABOND® PTS / PTL Filters

⇨ The Ideal Tool for Breaking Emulsions

MACHEREY-NAGEL

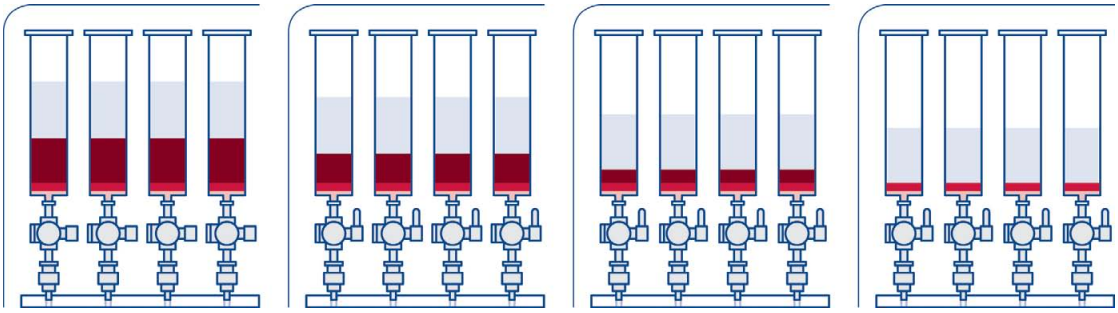
If you are working with LLE, PTS/PLT filters provide automatic separation of a two-phase mixture without a separation funnel.

- Two-phase mixtures are completely applied to the column
- Emulsions are eliminated as the denser phase passes through the membrane, but the lighter phase is trapped above
- Membrane automatically stops the flow when the denser phase has passed
- Uses gravity flow – no pressure or vacuum required
- Upper phase remains in the column, both phases are available for further analysis



PTS: For solvents that are **heavier** than water, e.g., for trichloromethane, dichloromethane. Max size 150 mL.

PTL: For solvents that are **lighter** than water, e.g., for diethyl ether, hexane. Max size 70 mL.



CHROMABOND® PTL in action: From left to right, PTL allows the aqueous phase (lower, red) through the membrane, and stops the organic phase (upper, colourless).

To process larger samples, or for greater automation of your sample preparation, ask us about DryDisk membranes for organic solvents that are denser than water.

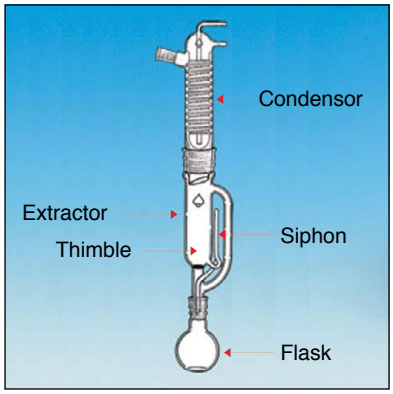
FREE SAMPLES are available!

Contact us for more information.

Soxhlet extraction is an effective method to use when your analytes have limited solubility and the impurity in your sample is insoluble in the desired solvent.

We offer quality supplies at the competitive prices you need!

- **Disposable Thimbles**
  - 18 to 80mm ID and 37 to 250mm height
  - Cellulose Thimbles - recommended for
    - Standard extraction methods including fats in food, vitamin A and carotene, lacquer and binder in paints, quality management of components used in pharmaceutical formulations, organic pollutants in tissues or soils.
  - Glass Fiber Thimbles - recommended for
    - Sampling dust particles and aerosols from gaseous streams up to 500°C
    - Extractions requiring solvents too aggressive for the cellulose thimbles
  - Quartz Fiber Thimbles - recommended for
    - Emissions testing in high-temperature environments up to 900°C
    - Testing acidic gases not compatible with micro-glass fiber thimbles

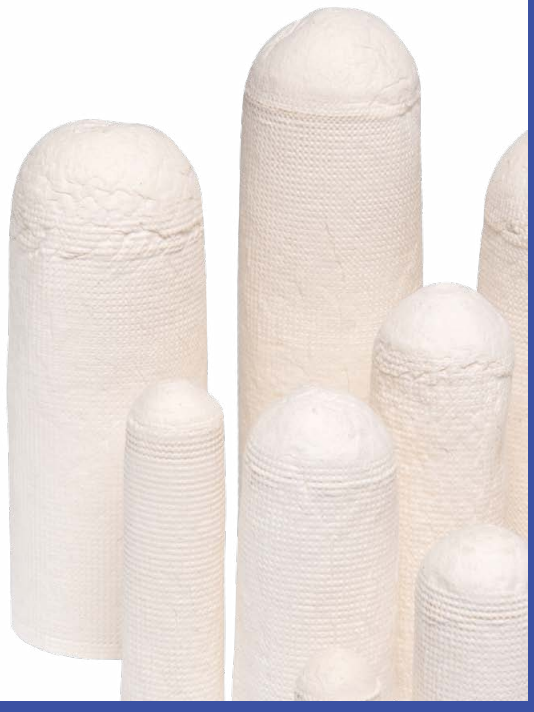


- **Reusable Glass Thimbles**
  - 28 to 45mm OD, 85 or 125mm height, and 6-160μm porosity
  - Cost effective only if cleaning thimble between uses is effective and not time consuming
- **Condensers**
- **Extractors**
- **Round bottom flasks**

Visit **www.chromspec.com/soxhlet** for more information.

Reduce Solvent Use

Micro Soxhlet extraction consumes less organic solvent. When limits of detection aren't crucial or sample size is limited, scale down to the 10 x 50mm thimbles and save money.



Economical Air and Water Volatiles Analysis

CDS 7000E Purge and Trap Concentrator

The CDS 7000 was acknowledged as a workhorse in the industry for years, now the 7000E delivers the same combination of qualities that made the 7000 such a popular choice, **but in a smaller and more cost effective package**.

- Proven, reliable performance
- Easy operation
- Best value / performance available
- Autosampler options include water only or soil / water

CDS Analytical, Inc.

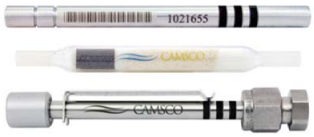
CDS 7300 / 7400 / 7500 Series Autosamplers

⇨ With modular towers, it is like having 3 systems in 1!

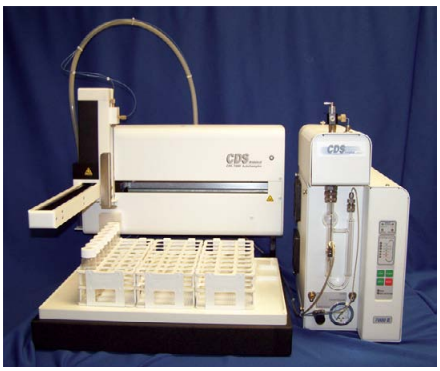
- CDS 7300 Purge & Trap Autosampler ⇨ ***Water only***
- CDS 7400 Purge & Trap Autosampler ⇨ ***Water / Soil / Dynamic Headspace***
- CDS 7500 Thermal Desorption Autosampler ⇨ ***Sorbent Tubes for Air Sampling***

With this modular platform, you can easily upgrade from the 7300 water-only autosampler to the 7400 or 7500 with a simple swap of towers. Changing from one mode to another can be accomplished in a matter of minutes!

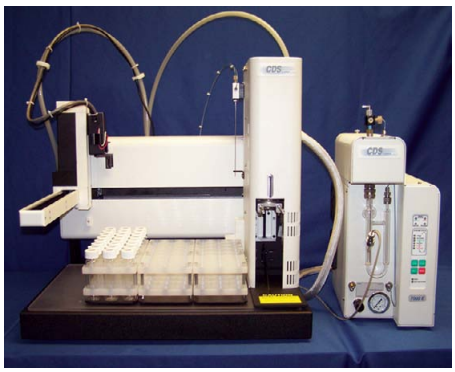
- 72 positions with removable sample trays for 40mL VOA vials or TD tubes
- Switch between water, soil and dynamic headspace modes in seconds
- Switching to thermal desorption takes only minutes
- Connects to existing CDS 7000E systems as well as other concentrators and/or desorbers on the market
- Performing multiple methods on one system saves significantly over the purchase of multiple systems
- Competitively priced



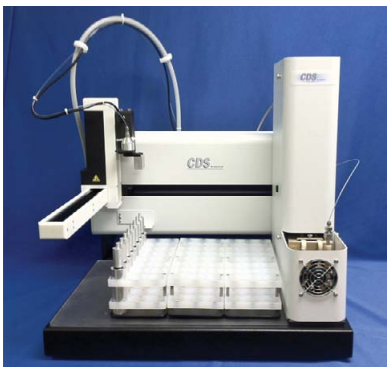
Visit
www.chromspec.com/camsco
for information on Camsco Thermal Desorption Tubes.



CDS 7300 (Water only)



CDS 7400 (Water & Soil)



CDS 7500 (TD Tubes)

Contact our Instrumentation Team for more information.

HTA HT2800T Autosampler

- ⇨ Static Headspace
- ⇨ SPME
- ⇨ Liquid Injection

} All-in-one Unit

- Lowest cost of ownership in the industry
- Superior thermal stability for SPME and headspace modes
- Touch screen control for exceptional ease of use
- 5+5 flexibility: 5 minutes to switch between modes, 5 minutes to switch between GC's
- Works on all GC systems and allows for dual injector configurations on most platforms
- Mounting design allows for injector maintenance without moving the autosampler
- Optional CFR 21 Part 11 compliant software

Smart Technologies for the best user experience

The HT2800T features a unique portfolio of patented, proprietary or licensed technologies:

- System integrity and vial leakage check for headspace analysis
- Syringe ID: a proprietary technology based on RFID tag to prevent mismatch errors
- Six-position oven that allows the optimization of preparation times for headspace and SPME

Contact our Instrumentation Team for more information.

Our professional in-house Instrumentation Specialists are available to provide you with a full range of services, including new product installation, on-site service and training, toll-free telephone support...and much more.

- Turnkey Systems
- Customization
- Automation
- Process Integration
- Installation
- Maintenance and Repairs
- Consultation and Training

We offer a solutions-based approach for chromatography and sample preparation instruments

Contact us for a FREE Consultation

CHROMSPEC™ UV Syringe Filters

Your best option for quality syringe filters at lower prices!

- Solvent resistant housing
- Made with North American membranes
- Sterile or non-sterile options
- GMF pre-filter option
- Pore sizes of 0.45µm and 0.22µm for HPLC and UHPLC applications
- 4mm, 13mm, 25mm and 30mm diameters
- Available membranes include: Glass Fibre, Mixed Cellulose Esters, Nylon, PES, Polypropylene, PTFE, PVDF and Regenerated Cellulose

NON STERILE
- Great performance for high throughput labs
- Ideal for HPLC-UV and GC-FID applications

STERILE
- ETO Sterilized and individually sealed to maintain sterility
- Ideal for sterile filtration and clarification

Choosing the right CHROMSPEC™ UV Syringe Filter

Nylon
- Hydrophilic, good solvent resistance and medium protein binding
- Filtration of all aqueous samples and most organic solvents
- Not recommended for acids

PTFE
- Highest solvent resistance and high protein binding
- Filtration of non-aqueous or solvent based samples
- Recommended for strong acids and bases

PVDF
- Highly resistant to most solvents and low protein binding
- General filtration of biological samples
- Filtration of all aqueous and most solvent based samples
- Filtration of proteins and tissue cultures

Mixed Cellulose Esters (MCE)
- Hydrophilic and very low protein binding
- Ideal for aqueous based samples, tissue culture and sensitive biological samples
- Low chemical resistance

PES
- Hydrophilic and low protein binding
- Ideal for aqueous based samples
- General filtration of biological samples

Polypropylene
- Hydrophobic
- High solvent resistance
- Low protein binding
- General filtration of biological samples
- Filtration of all aqueous and most solvent based samples

Regenerated Cellulose
- Hydrophilic
- Resistant to most solvents and aqueous solutions (pH range 3 -12)

Pre-Filters
- Recommended for removing particles in suspension
- Helps prevent clogging of the syringe filter membrane

Sterile
- Applications include: Cell culture media prep, mycoplasma removal, sterile filtration, including DMSO

Size Selection	
Syringe Filter Size	**Sample Volume**
4mm	2mL
13mm	6mL
25mm	70mL
30mm	90mL

Visit **www.chromspec.com/UV** for more information.

TRY IT NOW! **FREE SAMPLES are available!**

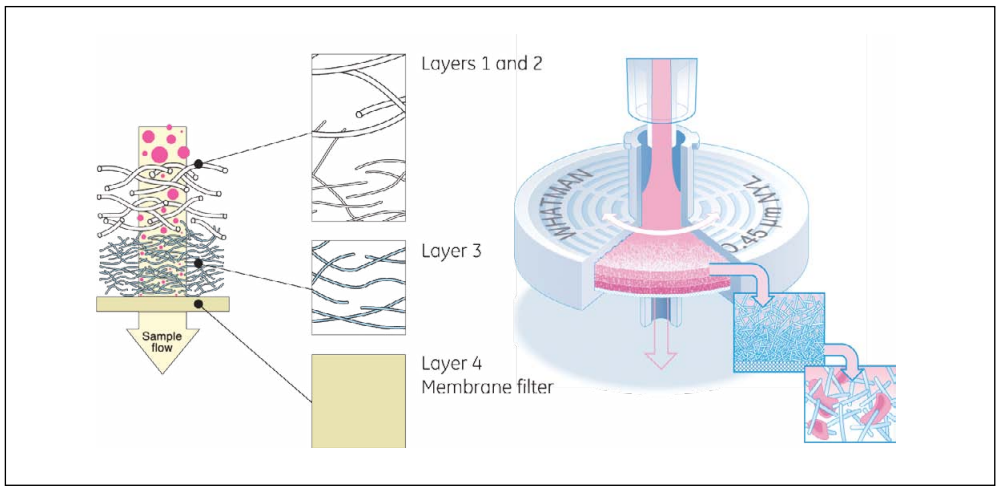
CHROMSPEC™…The Name for Affordable Quality

Whatman GD/X™ Syringe Filters

⇨ For samples with high solids content

Whatman GD/X™ syringe filters with integrated layered pre-filters are designed for exceptional loading capacity with high flow rates for thick, viscous samples.

- Integrated three-stage glass fibre pre-filter (10 µm to 0.7 µm) removes more solids without clogging
- Clean, inert and rugged polypropylene housing
- Pore sizes of 0.2 µm, 0.45 µm and 5 µm
- 13 mm or 25 mm diameters
- Available membranes include: Nylon, PES, Polypropylene, PTFE, PVD, Regenerated Cellulose and Cellulose Acetate



GD/X™ filters process three to seven times more sample volume than filters without a pre-filter.

Thomson SINGLE StEP Filter Vials

Reduce waste and improve speed and efficiency when filtering samples

- Easy to use: simply add your unfiltered solution to the vial base, squeeze the filter plunger into the base, and your vial is ready for the autosampler.
- Incorporates vials, caps, syringes, and filters into one unit
- Safer than syringe filters
  - Vial design limits pressure on membrane – less risk of bursting
  - Fully contained sample eliminates dangerous aerosols even if a filter bursts
- Available in Nylon, PES, PTFE and PVDF membrane materials

Is your analyte non-volatile, won't pass through a GC, and hard to detect using HPLC?

Derivatization reagents are designed to simplify chromatography by increasing sample volatility, allowing for simple GC-FID detection; or enhance UV-Visible absorption for routine HPLC detection.

EXPERT ADVICE

Contact our Technical Support Team for assistance in determining the ideal derivatization reagent for your particular analysis.

Chromatographic Specialties Inc. carries reagents from...

REGIS® TECHNOLOGIES, INC.

UCT PARTNER

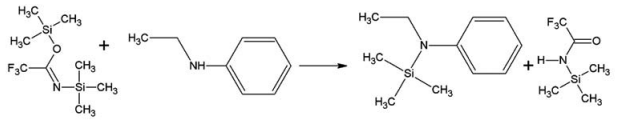
Thermo SCIENTIFIC (former Pierce products)

MN MACHEREY-NAGEL

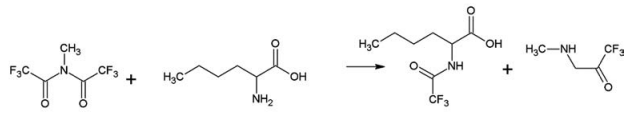
PICKERING LABORATORIES (Post-column Instruments too)

General Reagent Categories

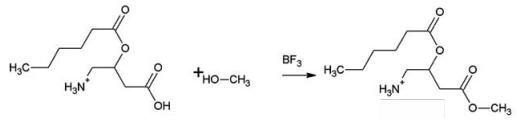
Silylation Reagents
- Mask polar groups with an alkyl-silyl group
- Improve volatility
- Additional carbon from alkyl groups increase FID response
- React with Alcohols, Phenols, Carboxyls, Amines and Amides



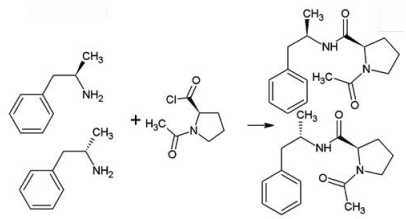
Acylation Reagents
- Mask polar groups with an acyl group
- Highly reactive, suited for analytes where steric hindrance may be a factor
- All acylation reagents add carbons & increase FID response
- Fluorinated reagents increase electron capture, for extremely sensitive ECD methods
- Ideal for highly polar, multi-functional compounds, such as carbohydrates and amino acids



Alkylation Reagents
- Reduce polarity by replacing active hydrogens with an alkyl group
- Fast, stable and quantitative
- Increase volatility and FID response
- Modify compounds such as carboxylic acids and phenols
- Commonly used to form esters
- Ether and amides also possible



Chiral Analysis of Amphetamines
- Regis' TPC reagent is ideal for fast and easy identification of amphetamine isomers
- 30 minute prep time creates diasteromers for FID analysis on standard GC columns



HPLC Derivatization
- We have instrumentation and supplies for pre- and post-column derivatization to add chromophores for improved HPLC detection

SPME, invented by Dr. Janusz Pawliszyn at the University of Waterloo, is a green sample preparation technique that provides repeatable results and is easily automated. Applications include food analysis, environmental samples, biologicals, in-vivo analysis, and more... the list continues to grow.

How does SPME work?

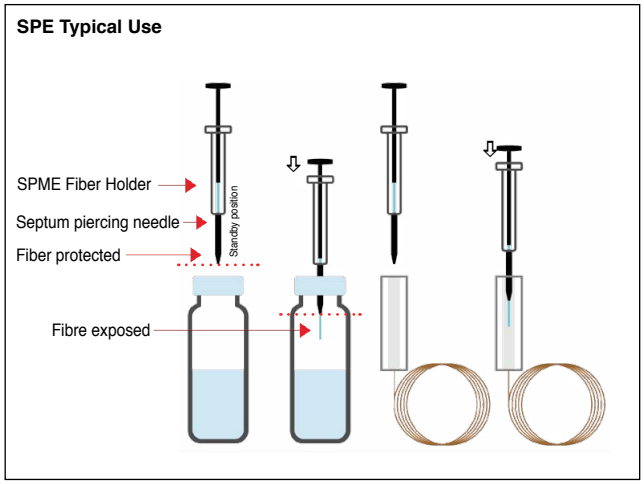
Step 1: Ready to sample. Fiber is withdrawn into needle for protection during handling and septum penetration.

Step 2: After penetrating sample vial septum, fiber is exposed for absorption by depressing plunger. Absorption can be from headspace (as shown) or direct from a liquid sample.

Step 3: Fiber is withdrawn into needle, ready for GC injection.

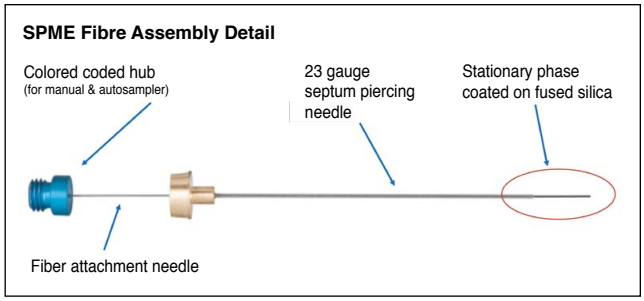
Step 4: Injection. Fiber is exposed for thermal desorption in GC inlet.

SPME requires minimal sample handling, saving operators time and laboratory space.



Restek PAL SPME Fibers

- Reliable performance that meets or exceeds the current market products
- Robust aluminum hub is more durable than plastic hubs
- Colour coding of hubs follows existing industry practice
- Fibers are 10 mm long, housed in a 23-gauge needle, and have a stationary phase bonded to a fused silica fiber



Which fiber is best for my application?

Choose stationary phases and film thickness for your application based on the properties of the compounds to be analyzed.

Target Analyte	Molecular Weight*	Recommended Fiber	Hub Colour
Nonpolar	125-600	7 µm polydimethylsiloxane (PDMS)	Green
Nonpolar, semivolatile	80-500	30 µm polydimethylsiloxane (PDMS)	Golden
Volatile	60-275	100 µm polydimethylsiloxane (PDMS)	Red
Polar, semivolatile	80-300	85 µm Polyacrylate (PA)	Grey
Highly volatile	30-225	95 µm Carbon wide range (WR)/PDMS	Dark blue

* These molecular weight ranges are a reasonable approximation; however, users should verify suitability for their specific application.

Coming Soon!

Restek SPME Arrow

- Optimized probe geometry and materials
- Lifetime is typically 2-3 times longer that classic SPME fibers
- 10 times greater sensitivity by providing 15 times more stationary phase than classic SPME fibers

Visit **www.chromspec.com/spme** for more information.