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Simplify the search for controlled substances in food with Agilent SampliQ SPE products

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Researchers working in the food safety area face numerous challenges when developing quantitative analysis for target contaminants, most significantly:

- A wide variety of matrices
- Low maximum residue levels (MRLs)
- The number and diversity of regulated chemicals

Solid-phase extraction (SPE) helps to provide a cleaner sample and is therefore a cornerstone of these complex sample analyses. Even when using highly specific detectors such as LC/MS/MS, SPE is critical because ion suppression from co-eluting impurities can adversely affect precision and accuracy. A cleaner extract can mean:

- Less complicated analysis conditions
- Longer HPLC column life
- More accurate, reproducible results

Three polymer compounds provide the clean samples you need

Agilent offers three cartridges with differing resin compounds to provide an excellent [SPE solution](#) for the widest possible range of matrices and target compounds. **Figure 1** provides quick guidance on selection of the appropriate polymer cartridge. But a more in-depth discussion of each cartridge’s advantages is worthwhile.

Agilent SampliQ OPT covers a wide range of monitored compounds

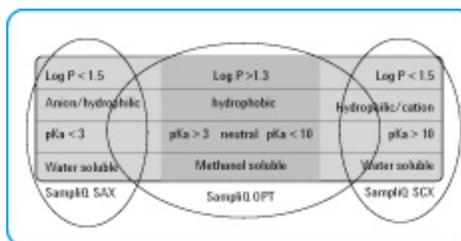


Figure 1. Guide to selection of SampliQ polymer sorbents. ([Click here](#) to see this image larger.)

An Optimized Polymer Technology (OPT) that incorporates a novel polyamide chemistry, this resin exhibits retention for both polar and non-polar compounds based on their hydrophilic/lipophilic character. Its reversed phase behavior provides ease of method development and compatibility with both gas chromatographic and liquid chromatographic separations of the extracts [1]. As a general rule, SampliQ OPT will retain hydrophilic compounds with log P values above 1.5, hydrophobic compounds, acidic compounds with pKa's above 3 and basic compounds with pKa's below 10. This covers a wide range of compounds monitored by the food industry including pesticides, antibiotics, endocrine disruptors and many other classes.

Agilent SampliQ SCX provides superior cleanup when analyzing basic compounds.

A sulfonic acid modified divinyl benzene polymer, this resin should be used when the target analytes are cationic, basic compounds (pKa > 8). SampliQ SCX is a mixed mode resin which exhibits retention for both basic and neutral compounds over a wide range of pKa's and hydrophylicity (log P). A simple two-step elution method provides a means to separate the neutral compounds in one eluent and the basic compounds in a second eluent [2], which is important when the target analytes have significantly different chemical properties. The two-step elution process also means that the resin provides superior clean-up of complex samples when the target analytes are basic compounds. The resin is inert to a wide variety of solvents, is stable in pH ranges 0-14 and is water-wettable.

Agilent SampliQ SAX is the right choice for analyzing anionic, acidic compounds.

A tertiary amine modified divinyl benzene polymer, this resin should be used when the target analytes are anionic, acidic compounds. Like the other SampliQ polymer resins, this resin has a mixed mode behavior which retains both acidic and neutral compounds over a wide range of pKa's and hydrophylicity (log P). A simple two step elution method results in recovery of neutral compounds in one fraction and the acidic compounds in the second fraction [3].

4-step method development assures superior results

Method development is faster with polymer resins because these resins retain such a wide variety of chemical classes. And a simple method is frequently all that is necessary for the optimal use of these single-use cartridges.

An additional advantage of polymer resins over silica based resins is the fact that the retention and recovery of analytes is unaffected if the resin should be accidentally dried during the conditioning process.

There are typically four steps in the SPE method development process as shown in **Figure 2**:

1. Conditioning
2. Loading
3. Washing
4. Elution

SampliQ OPT uses a water-miscible, organic conditioning solvent to prepare (wet) the surface to receive the sample. The next step in the conditioning process is to remove the methanol with at least 5 bed volumes of water.

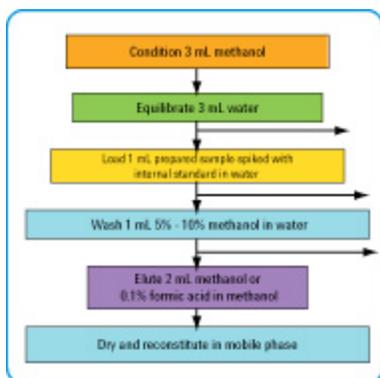


Figure 2. Method development process for SampliQ OPT, 3 mL cartridge. ([Click here](#) to see this image larger.)

Then the prepared sample is loaded as an aqueous solution after spiking with an internal standard. Vacuum may be required depending on the viscosity of the sample, and flow should be between 1 and 2 mL/min. A 60mg bed of resin will perform comparably to a 200mg bed of C18 silica sorbent.

The washing step should use the strongest (highest % organic) solvent that will not elute the target compounds. A volume equivalent to a minimum of 5 times the bed volume should be used. A brief dry of the cartridge should be performed to remove as much residual water as possible at this step. During initial method development, the wash should be analyzed to confirm that no breakthrough of target analyte occurred.

The elution step should use the weakest (lowest % organic) that will elute the target compounds. A recommended volume for the elution step is 1-2 mL for a 3 mL cartridge. The eluate is collected, then evaporated to dryness. The sample should be brought to the desired volume in water or starting mobile phase solution.

Performance is outstanding in both “wet” and “dry” conditions

With some types of SPE cartridges significant loss of sample occurs if the cartridge is dried between the methanol conditioning and water equilibration steps. With the polymer SampliQ products, if the cartridge accidentally runs dry there is no difference in performance. **Figure 3** shows the extraction recoveries performed under two different experimental conditions. In the “wet” experiment, the cartridge is conditioned with methanol (MeOH) and the equilibration solvent (water) is added before the cartridge goes dry. In the “dry” experiment, the cartridge is conditioned with MeOH but the cartridge is dried under vacuum for 10 minutes before the addition of water for the equilibration step. Notice that there is no difference in recovery observed when the cartridge is dried in the pre-extraction steps and when it is not. The percent relative standard deviations (% RSD) of the recoveries for each of the compounds is less than 5% (n=5) for samples spiked at 10 ng/mL.

Get more out of SPE with Agilent SampliQ polymer resin cartridges

For researchers faced with the challenges of multi-residue, trace chemical analysis in a variety of matrices, the Agilent SampliQ polymer sorbents offer:

- Ease of method development
- Mixed mode retention mechanisms
- Excellent reproducibility
- Rigorous manufacturing quality control
- High recoveries even when dried

Learn more about how you can extract the clean samples you need for outstanding results with Agilent’s range of [SampliQ polymer resin cartridges](#). Or access specific solutions in the referenced Application Notes.

References

1. Carol Haney Ball, “Agilent’s New Polymer Solid-Phase Extraction Cartridges: SampliQ OPT”, Agilent Application Note [5989-8869EN](#), **2008**
2. Carol Haney Ball, “Agilent’s New Mixed-Mode Cation Exchange Polymer Solid-Phase

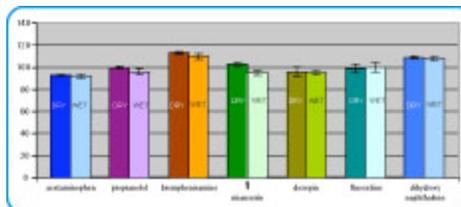


Figure 3. Recovery study for “wet” and “dry” conditions, using SampliQ OPT. ([Click here](#) to see this image larger.)

Extraction Cartridges: SampliQ SCX", Agilent Application Note [5989-8945EN](#)

3. Carol Haney Ball, "Agilent's New Mixed-Mode Anion Exchange Polymer Solid-Phase Extraction Cartridges: SampliQ SAX", Agilent Application Note [5989-8944EN](#)