INTip™ Solid Phase Extraction
Dispersive Pipette XTRaction Product Guide
INTip™ SPE utilizes a patented technology known as Dispersive Pipette XTRaction. This device is unique from all other SPE devices because sorbent is loosely contained within a pipette tip.

What is Dispersive Pipette XTRaction?
This technology enables solid phase extraction inside a pipette tip for easy sample preparation. XTR tips contain loose sorbent between a top barrier and bottom frit. The disperser helps to perturb the sample solution and loose sorbent during aspirate and dispense steps. This mixing provides a highly efficient interaction of the sorbent with analytes of interest resulting in ideal analyte recoveries. Our tips can be used for cleanup or bind-wash-elute protocols for a wide variety of sample preparation applications.

Technology Advantages:
- Easy to use, easy to automate
- Consistently high recoveries
- Rapid extraction times
- Seamless integration with any workflow or method
- Custom method development
- No positive pressure/vacuum manifold

XTR Tip Anatomy:

XTR Tips:
- Manufactured to order for customization
- Can hold 1 - 100 mg of sorbent (depending on format)
- Variety of sorbent chemistries available
- Available in manual, semi-automated and fully automated formats

μXTR Tips:
- Same technology advantages
- Proprietary design that utilizes a unique low-retention frit
- Compatible with all DPX sorbents with > 50 μm particle size
- Can hold as low as 1 mg of sorbent
- Eliminate solvent evaporation/dry down step
- Increase sensitivity
- Reduce elution volumes to as low as 25 μL for up to 8X concentration factor
- Available in Hamilton or Integra 300 μL formats

XTR Tip Formats:
- Hamilton - 300 μL, 1 mL
- Integra - 300 μL and 1250 μL
- Gerstel - 1 mL
- Tecan - 200 μL, 1 mL
- Eppendorf - 1 mL
- Manual - 1 mL, 5 mL
DPX offers sorbent chemistries in a range of phases to capture unique selectivity for a diverse spectrum of analytes.

**Sorbent Phase Selection Guide**

<table>
<thead>
<tr>
<th>Acidic (pKa &lt; 1)</th>
<th>Hydrophilic</th>
<th>Hydrophobic</th>
<th>Hydrophilic</th>
<th>Basic (pKa &gt; 10)</th>
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<tbody>
<tr>
<td>WAX</td>
<td>SAX</td>
<td>C18</td>
<td>SX</td>
<td>WCX</td>
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</table>

**Weak Anion Exchange - WAX**
Our weak anion exchange (WAX) sorbent has a secondary amine phase with a styrene divinyl benzene backbone for the binding of strong and weak acids.
Particle Size Ranges: 10-20 µm and 55-65 µm  
Pore Size: 80 Å  
Capacity: 800-1300 µmol/g  
pH Range: 0-14

**Weak Cation Exchange - WCX**
Our WCX sorbent has a carboxylic acid stationary phase with a styrene divinyl benzene backbone for the binding of strong and weak bases.
Particle Size Ranges: 55-65 µm  
Pore Size: 80 Å  
Capacity: 800-1200 µmol/g  
pH Range: 0-14

**Strong Cation Exchange - SCX**
Our SCX sorbent has a sulfonated stationary phase with a styrene divinyl benzene backbone for the binding of basic compounds.
Particle Size Ranges: 10-20 µm, 25-35 µm and 55-65 µm  
Pore Size: 80 Å  
Capacity: 800-1300 µmol/g  
pH Range: 0-14

**Strong Anion Exchange- SAX**
Our SAX sorbent has a quaternary amine phase with a styrene divinyl benzene backbone for the binding of acidic compounds.
Particle Size Ranges: 50-65 µm  
Pore Size: 80 Å  
Capacity: >65 µmol/g  
pH Range: 0-14
Each sorbent’s mass can be customized for optimal extraction characteristics for any analyte/matrix combination.

**Sorbent Phase Selection Guide**

**Silica**
Our silica sorbent is amenable to extraction via hydrogen bonding.

- Particle size: 40-63 µm
- Pore size: Diameter of 60 Å
- Surface area: 500-600 m²/g
- pH range: 6.5-7.5

**C18**
Our C18 sorbent has a silica gel backbone. It is endcapped, with a carbon load of 18-22%.

- Particle size ranges: 40-63 µm
- Pore size: 60 Å
- Surface area: 500-600 m²/g

**Reverse Phase - RP**
Our RP sorbent is a styrene divinylbenzene polymer with an average particle size of 75 µm. It is ideal for nonpolar and slightly polar compounds (logP > 1.8).

- Particle size: avg. 75 µm
- Pore size: 150 Å
- Surface area: 900 m²/g

**HybridSPE®**
A proprietary zirconia coated silica that selectively removes phospholipids from biological samples. Unique chemistry for excellent recovery of both hydrophobic and hydrophilic analytes.

*Please see Supelco/Sigma website for ordering information*

**Mixed Mode**
Our mixed mode products are completely customized by choosing any of the above chemistries to combine in one tip. Each sorbent’s mass can also be customized for optimal extraction characteristics for any analyte/matrix combination.

Ex. WAX/RP, WAX/SCX
Dispersive Pipette XTRaction provides a low cost INTip™ solution to isolate the analyte of interest.

Solid Phase Extraction
Traditional SPE workflows include conditioning the sorbent, exposing the sorbent to the sample to bind the analytes of interest, washing excess sample/matrix off the sorbent and then eluting the analyte in a clean solution. The same process is performed with XTR tips using a series of aspirate and dispense steps as shown below.

Replace your current SPE
• Ideal when looking for high throughput, automation and ease of use

Improve your current SPE
• Resolve current issues with a DPX solution
• Reduce sample volume, internal standard, evaporation time

Bind-Wash-Elute Workflow

Disperser forces turbulent mixing of loose sorbent

Condition

Bind

Wash

Elute

Isolate analytes of interest

Analytes bind to sorbent

Remove matrix interferents

Greater recoveries with a customized mixed mode product

Product Customization for Oral Fluid Extraction
Cation exchange sorbent (SCX) is routinely used to extract a comprehensive panel of therapeutic and abused drugs from oral fluid. While SCX provides high recoveries of most of the compounds of interest, it does not provide high recoveries of barbiturates, as shown in the figure to the left. Unlike all other SPE companies, DPX’s unique ability to customize products allowed the incorporation of weak anion exchange sorbent (WAX) along with the SCX sorbent in the same XTR tip to significantly improve barbiturates recoveries.
Cleanup with Dispersive Pipette XTRaction

**Extract and remove matrix**

Get the best compromise of cleanliness and minimal sample preparation time with XTR cleanup.

- Rapid extraction times
- Easy sample cleanup

**Cleanup**

XTR tips can house sorbents that provide selective removal of matrix interferents. Cleanup takes seconds, only the time for an aspirate and dispense step. Ex. HybridSPE sorbent

**Workflow**

Clean extract ready for analysis

Aspirate Sample/Sorbent

Mix via aspirate/ dispense steps to bind matrix interferents

Transfer analyte rich upper acetonitrile layer

Clean extract ready for analysis

**DPX + SALLE**

XTR tips can house sorbents like Weak Anion Exchange (WAX) or Reverse Phase (RP) for cleanup and the salt (S) necessary for SALLE (Salting-out Assisted Liquid-Liquid Extraction). This methodology is ideal for applications that require acetonitrile for protein precipitation and are focused on hydrophobic analytes. Ex. WAX-S, RP-S

**Vitamin D Extraction Using WAX-S**

Extraction efficiency was 90% or greater for 25-hydroxyvitamin D2 (25OH2) and 25-hydroxyvitamin D3 (25OH3) from serum using a Tip-on-Tip protein precipitation method and XTR tips with WAX-S sorbent for cleanup.
Increase versatility - Dispersive Pipette XTRaction can be used for a variety of applications and with a variety of matrices.

Matrix Flexibility

- Urine
- Blood/Serum
- Oral Fluid
- Tissue
- Meconium
- Hair
- Water
- Food Samples
- Plant Samples (Ex. Cannabis)

Clinical Applications:

- Comprehensive Drugs of Abuse Panels, Steroids, Hormones, Vitamin D

Forensic Applications:

- Cannabinoids, Opiates, Benzodiazepines

Food Safety Applications:

- Multi-residue Pesticides, Aminoglycoside Antibiotics, β-agonists, Melamine, Cannabinoids

Application notes are available for download on our website. Below is a small list of developed methods. Click on any link to download the application note directly from our website.

RP

- Analysis of Potency in Cannabis Material
- Catecholamines and Metabolites in Urine
- Pesticides in Fruits and Vegetables

WAX

- Analysis of Benzodiazepines in Biological Samples
- Pesticides in Food Samples with High Fat Content

DPX + SALLE

- THC in Urine
- THC in Blood
- Vitamin D from Serum
- Cortisol in Urine and Saliva
- Benzodiazepines in Meconium

WCX

- Rapid Analysis of Aminoglycoside Antibiotics in Bovine Tissues

HybridSPE®

- Analysis for the Measurement of Nestorone® in Human Serum
- Analysis for the Measurement of Male Drug Hormones in Human Serum

Mixed Mode

- Comprehensive Extraction of Drugs/Metabolites in Urine
- Comprehensive Extraction of Drugs/Metabolites in Oral Fluid

Contact us if you have any questions about specific applications of interest.
Need custom method development? Our application scientists can help integrate DPX products into your existing workflow or develop new methods.

## General SPE Guidelines

This information is provided as a starting point for method development - optimized conditions may vary.

Table 1. Correlation of sample solution volume, sorbent amount, and volume of wash and elution solutions. Elution volumes depend on the chemical nature of the analyte, its relative concentration in the matrix, the chemistry of the solution solvent and the sorbent bed mass.

* Sample Solution = total volume of sample after any pretreatment; including, but not limited to, hydrolysis

<table>
<thead>
<tr>
<th>Sample Solution Volume</th>
<th>Sorbent Amount</th>
<th>Wash + Elution Volumes</th>
</tr>
</thead>
<tbody>
<tr>
<td>≤200 μL</td>
<td>3 mg</td>
<td>≥150 μL</td>
</tr>
<tr>
<td>200-400 μL</td>
<td>6 mg</td>
<td>≥300 μL</td>
</tr>
<tr>
<td>400-600 μL</td>
<td>10 mg</td>
<td>≥500 μL</td>
</tr>
<tr>
<td>&gt;600 μL</td>
<td>20 mg +</td>
<td>≥800 μL</td>
</tr>
</tbody>
</table>

Table 2. Correlation of organic solvent percentage in the DPX procedure based on analyte polarity.

If the method has analytes of various polarities, base the protocol on the most polar analyte for the sample and wash conditions, but the lowest polarity analyte for the elution conditions. For example, a comprehensive method would have 10% organic in the sample and wash solution, but would elute with 100% organic solvent.

<table>
<thead>
<tr>
<th>Analyte Polarity</th>
<th>% Organic in Sample</th>
<th>% Organic in Wash</th>
<th>% Organic in Elution</th>
</tr>
</thead>
<tbody>
<tr>
<td>High (Polar)</td>
<td>&lt;10%</td>
<td>&lt;10%</td>
<td>≥30%</td>
</tr>
<tr>
<td>Medium</td>
<td>&lt;20%</td>
<td>&lt;20%</td>
<td>≥50%</td>
</tr>
<tr>
<td>Low (Non-Polar)</td>
<td>&lt;50%</td>
<td>&lt;50%</td>
<td>100%</td>
</tr>
</tbody>
</table>