







Suggested Solid Phase Extraction method using SiliCycle[®] SiliaPrep[™] Strong Cation Exchanger (SCX)

Supported sulfonic acids are in a class of strong acids ($pK_a^* < 1$) widely used in different fields of synthetic organic chemistry. The applications are widely known and the various form of the products are used in a large number of settings going from drug discovery laboratories up to the manufacturing processes. The most common application is probably the use as strong cation exchanger (SCX) for the "Catch and Release" purification of amine in SPE cartridges. We describe here a generic sample preparation method using **SiliaPrep[™] Strong Cation Exchanger (SiliaPrep[™] SCX (Tosic Acid) and SCX-2 (Propylsulfonic Acid))** for the extraction of cationic analytes from aqueous solution. The retention mechanism is based on the fact that both **SiliaPrep[™] SCX** and **SCX-2** maintain a negative charge throughout the pH scale compared to the basic analyte that can be positively charge depending on the solution pH.

This is a recommended sample preparation method, however this method has not been verified in SiliCycle[®] laboratories but is based on methods used with similar matrices and analytes. Therefore, before trying the procedure directly on the matrix, you should first develop a procedure using pure solvent containing the analyte. You will find below, tips for the choice of solvent that you should use to establish your SPE method development:

* The pK_a represents the pH at which the proportion of protonated and deprotonated molecules is equal (1:1).

EXTRACTION PROCEDURE

 SPE Cartridge type:	<ul style="list-style-type: none"> ● SiliCycle[®] SiliaPrep[™] SCX (PN: SPE-R60530B) ● SiliCycle[®] SiliaPrep[™] SCX-2 (PN: SPE-R51230B) <p>Note: Consult SiliCycle[®] catalog for the choice of cartridge volume and sorbent bed weight availability.</p>
 Analyte structure:	<ul style="list-style-type: none"> ● Cationic analytes (ie: 1°, 2° and 3° amine) <p>Note: Use Weak Cation Exchanger (WCX) (SiliaPrep[™] Carboxylic Acid (CAA)) for the extraction of quaternary amine (4°)</p>
 Sample pre-treatment:	<ul style="list-style-type: none"> ● Sample solution pH adjustment <p>Tips:</p> <ul style="list-style-type: none">  Solution pH neutral or acid ($pH < 7$) or  Two pH units less than analyte pK_a <p>Procedure:</p> <ul style="list-style-type: none">  Adjust the sample solution pH using buffered solution to reach the pH at wich the analyte is all charged (related to the pK_a) <p>Precautions:</p> <p>The salt concentration should not be too high (< 30-50 mM). Also, the analyte retention strength is related to the facility of the counter ion to be replaced by the sorbent counter ion (SiliaPrep[™] SCX and SCX-2 have an hydrogen counter ion). See the chart below.</p>

<p> Sample pre-treatment (cont):</p>	<table border="1" style="width: 100%; text-align: center;"> <thead> <tr> <th style="background-color: #000080; color: #FFD700;">Anions</th> <th style="background-color: #000080; color: #FFD700;">Strength</th> <th style="background-color: #000080; color: #FFD700;">Cations</th> </tr> </thead> <tbody> <tr> <td></td> <td style="vertical-align: middle;">Weaker</td> <td>Li</td> </tr> <tr> <td>OH</td> <td rowspan="13" style="vertical-align: middle;"> </td> <td>H</td> </tr> <tr> <td>F</td> <td>Na</td> </tr> <tr> <td>Acetate</td> <td>NH₄</td> </tr> <tr> <td>Formate</td> <td>K</td> </tr> <tr> <td>HPO₄</td> <td>Mg</td> </tr> <tr> <td>HCO₃</td> <td>Fe</td> </tr> <tr> <td>Cl</td> <td>Ca</td> </tr> <tr> <td>Br</td> <td>Cu</td> </tr> <tr> <td>Citrate</td> <td>Pb</td> </tr> <tr> <td>Tosylate</td> <td>Ag</td> </tr> <tr> <td></td> <td>Stronger</td> <td>Ba</td> </tr> </tbody> </table> <p style="text-align: center;">Chart 1: Counter ion strength</p>	Anions	Strength	Cations		Weaker	Li	OH		H	F	Na	Acetate	NH ₄	Formate	K	HPO ₄	Mg	HCO ₃	Fe	Cl	Ca	Br	Cu	Citrate	Pb	Tosylate	Ag		Stronger	Ba
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Br		Cu																													
Citrate		Pb																													
Tosylate		Ag																													
		Stronger	Ba																												
<p> SPE extraction using SiliaPrep™ SCX or SCX-2 :</p>		<ul style="list-style-type: none"> <p> Column conditioning step</p> <ul style="list-style-type: none"> <u>Solvent</u>: Methanol, acetonitrile or THF (1-2 column volume) <u>Flow rate</u>: 2-4 mL/min <p> Column equilibration step</p> <ul style="list-style-type: none"> <u>Solvent</u>: Buffered solution used for the sample preparation (1 column volume) <u>Flow rate</u>: 2-4 mL/min <p> Sample loading</p> <ul style="list-style-type: none"> Load directly the prepared sample on the top of the cartridge <u>Flow rate</u>: same rate as the cartridge volume ⇒ 1 mL SPE Cartridge : 1 mL/min ⇒ 3 mL SPE Cartridge : 3 mL/min ⇒ 6 mL SPE Cartridge : 6 mL/min, etc. <p> Interferences washing step</p> <ul style="list-style-type: none"> <u>Solvent</u>: Buffured solution (1-2 column volume) <u>Flow rate</u>: 1-2 mL/min <p>Note: For lipophilic interferences washing, add 10-20 % of methanol or acetonitrile to the buffer</p> <ul style="list-style-type: none"> <p> Analyte elution step</p> <ul style="list-style-type: none"> <u>Solvent</u>: Strong base in polar organic solvents and/or used a high concentration of strong counter ion as displacer (2-4 column volume) <u>Flow rate</u>: 1 mL/min <p>Note: Drying step (under vacuum) can be done before the analyte elution step. For weak cations elution, use solvent with a pH adjusted to 2 units above the pK_a of the analyte (to neutralize the analyte charge).</p>																													
<p> Analytical method :</p>		<ul style="list-style-type: none"> Related to the application. 																													
<p> General comments :</p>	<ul style="list-style-type: none"> For the "Catch and Release" purification of amine in organic solvent like methanol, refer to the SiliaPrep™ Propylsulfonic Acid section in the SiliCycle® Catalog. 																														