Ion Exchange Sorbents for Metals Extraction- Analysis & Sorbent Use Selection Guide

UCT ENVIRO-CLEAN® (Ion-Exchange Cartridges) *

The determination of trace metals in aqueous samples or other matrices often require sample pre-treatment and cleanup procedures prior to analysis by using specific ion-exchange sorbents. The sorbents are used to eliminate matrix interferences and achieve high concentrations of metal ions for good analytical accuracy. They are important when using such techniques as AA, IES and ICP-AES.

The use of ion-exchange sorbents for the preconcentration, separation and determination of metal ions for trace analysis is well established in the literature. Selection of an appropriate sorbent ensures both high efficiency in metal chelating while minimizing the mass of sorbent required for a particular analytical task. A high efficiency sorbent means that a smaller bed mass may be used thereby reducing the quantity of solvent required for elution yielding greater analytical sensitivity.

Recommendations in this application note include the following metal ions:

Zinc (II)	Arsenic (V)	Tin (IV)	Selenous (IV)
Mercury (II)	Chromium (III)	Copper (II)	Platinum (0)

Other metal ions may be extracted by the use of these ion-exchange sorbents

Sorbent Selection for Metals Extraction

Solid-phase sorbents have differing capacity and selectivity for various metal ions due to the specific nature of the ion-exchange functional group, the metal species and the valence state of the metal of interest. Depending on the specific metal ion of interest, elution of the cartridge may be most efficient using both the acid followed by the base elution procedure. This can be determined by looking at the following **Extraction Protocol Tables**. For example, when eluting Hg(II) from PSA the highest recovery is obtained using acid elution (green box) followed by base elution (yellow box).

Sample Analysis

1) **Sample Extraction**

   a) Assemble an all glass extraction apparatus

   b) Place a UCT ENVIRO-CLEAN® cartridge on the apparatus

   Note: Cartridge selection will depend on the volume of sample or the concentration of metal to be extracted

   c) Condition 1mL cartridge by adding 3 mL of methanol. (Larger cartridges will require a larger volume of solvent and water wash volume in steps c) and d)

   d) Add 3 mL of reagent water and allow to drip through the cartridge

   Note: Do not allow the cartridge to dry out after addition of water, otherwise repeat step d)

   Add 10-50 mL of sample water to the cartridge. A larger sample volume may be used depending on metal concentration or suspended solids content

   e) Adjust vacuum setting so that the water flows at 1-3 mL/minute until sample has passed completely through the cartridge

   f) Allow the cartridge to air dry for about 1 minute under full vacuum

2) **Elution--<u>Acid</u>**

   a) Prepare a 100 mM nitric acid elution solution

   b) Place a collection vial in the vacuum manifold

   c) Add 3 mL of the nitric acid solution to the cartridge

   d) Adjust flow rate for a flow of 1-3 mL/minute

   e) Dilute eluant to an appropriate volume for detection using reagent water

   f) Sample is ready for analysis

3) **Elution--<u>Base</u>**

   a) Prepare a 100 mM triethylamine elution solution

   b) Place a collection vial in the vacuum manifold

   c) Add 3 mL of the triethylamine solution to the cartridge

   d) Adjust flow rate for a flow of 1-3 mL/minute

   e) Dilute eluant to an appropriate volume for detection using reagent water

   f) Sample is ready for analysis

4) Analysis

a) Prepare calibration curves for use with atomic absorption (AA) or Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) using appropriate metals standards

Extraction Protocol Tables

How to use these tables: When choosing an ion-exchange sorbent to capture arsenic (V) for example, all ion-exchange sorbents will capture a small quantity of metal ions, however, only a base extraction would elute metal ions from these sorbents. For extraction of zinc ions, all sorbents would have moderate to high capacity but elution could only occur from the sorbent using acidic elution conditions.

Acid Extraction Protocol

Sorbent	Cu (II)	Zn (II)	As (V)	Sn (IV)	Se (IV)	Hg (II)	Cr (III)	Pt (0)
PSA	Green	Green	Red	Red	Green	Green	Yellow	Yellow
BCX-HL	Green	Yellow	Red	Red	Red	Green	Yellow	Red
CCX	Yellow	Yellow	Red	Yellow	Red	Green	Red	Green
TAX	Green	Green	Red	Yellow	Red	Green	Yellow	Yellow
THX	Green	Yellow	Red	Red	Yellow	Yellow	Red	Red
NAX	Green	Green	Red	Red	Red	Green	Red	Red

Base Extraction Protocol

Sorbent	Cu (II)	Zn (II)	As (V)	Sn (IV)	Se (IV)	Hg (II)	Cr (III)	Pt (0)
PSA	Red	Red	Yellow	Yellow	Green	Yellow	Red	Red
BCX-HL	Red	Red	Yellow	Yellow	Yellow	Green	Yellow	Red
CCX	Red	Red	Yellow	Green	Yellow	Yellow	Red	Red
TAX	Red	Red	Yellow	Green	Yellow	Green	Red	Red
THX	Red	Red	Yellow	Yellow	Yellow	Green	Red	Red
NAX	Red	Red	Yellow	Yellow	Yellow	Yellow	Red	Red

Green — Good to High Capacity
Yellow — Moderate Capacity
Red — Little or No Capacity

Ion-Exchange Sorbent Key

PSA	Primary secondary amine
BCX-HL	Benzene sulfonic acid –High Load
CCX	Carboxylic acid
TAX	Triacetic acid
THX	Sulfhydryl (Thiopropyl)
NAX	Aminopropyl

Primary Secondary Amine (PSA)

The PSA ion-exchange sorbent has a significant capacity for Hg(II) and Se(IV) followed by a lesser capacity for Sn(IV), Cu(II), Zn(II) and Cr(III). Metal ions are readily eluted from PSA by the use of weak acid solutions such as 100mM nitric acid solution. Additional recovery for selenium can be obtained by following the acid elution by the use of 100 mM triethylamine solution.

Benzenesulfonic Acid-High Load (BCX-HL)

The BCX-HL ion-exchange sorbent is the least selective of all ion-exchange sorbents and has significant capacity for Hg(II) and Sn(IV) thus ensuring high extraction efficiency for trace analysis. It is also a strong sorbent for Cu(II), Zn(II), Cr(III) and small amounts of Pt. In most cases, metal ions are readily eluted from BCX-HL by the use of 100mM nitric acid solution. Improvement in Hg(II) recovery yield and Sn(IV) can be achieved when eluting with 100 mM triethylamine solution.

Carboxylic Acid (CCX)

The CCX sorbents have high selectivity for Sn(IV) and Hg(II). Metal ions are readily eluted from CCX by the use of weak acid solutions such as 100mM nitric acid solution. Sn(IV) is eluted using 100 mM triethylamine solution. Additional Hg(II) is released under basic elution.

Triacetic Acid (TAX)

TAX sorbents have the highest affinity for Sn(IV) and Hg(II) followed by lesser amounts of Cu(II) and Zn(II). Metal ions are readily eluted from TAX by the use of weak acid solutions such as 100 mM nitric acid solution. Sn(IV) is eluted using 100 mM triethylamine solution. Additional Hg(II) is released under basic elution.

Sulfhydryl(THX/THIOPROPYL)

THX sorbents have the highest affinity for Hg(II) and Sn(IV), and approximately equal weights of Sn(IV) and Cu(II). Metal ions are readily eluted from THX by the use of weak acid solutions such as 100mM nitric acid solution. Sn(IV), Se(IV) and Hg(II) are eluted using 100 mM triethylamine solution.

Aminopropyl (NAX)

The NAX ion-exchange sorbent has a significant capacity for Hg(II) followed by Se(IV). Metal ions are readily eluted from NAX by the use of weak acid solutions such as 100mM nitric acid solution. Additional Hg(II) is released under basic elution.

For further data, specific information and discussion of each sorbent see separate UCT publications: **Topics in Solid-Phase Extraction: Metals Analysis.**

*UCT ENVIRO-CLEAN® Ion-exchange cartridges are available in a variety of cartridge sizes, sorbent mass and particle size for most analytical requirements. For further information, contact UCT.