Sample Identification and Information

NOTE: This worksheet may be used for up to four samples

Chemistry Project Number ____________________  Submitter _________________________________

Isotopes of Interest        Al  ____       Be  ____       Ca  ____       Cl  ____       I  ____

X  3  1  6  5  7

PRIME Lab ID  ________ ________ ________ ________
              Sample 1    Sample 2    Sample 3    Sample 4

User ID  ________ ________ ________ ________
         Sample 1    Sample 2    Sample 3    Sample 4

Sample Description and Comments

_________________________________________________________________________________
Sample 1
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
Sample 2
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
Sample 3
_________________________________________________________________________________
Sample Preparation

Date Started ______________________   _______   By

NOTE: Leach centrifuge tubes in 3% nitric acid solution for at least 1 hour prior to using.

NOTE: If chloride levels are not provided for the samples, determine the chloride concentration by Method AW0005, Determination of Chloride in Water Samples by Ion Selective Electrode.

Measure sufficient sample to produce 5 to 25 mg of silver chloride into a suitable container.

Sample amount

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
<th>Sample 4</th>
<th>By</th>
</tr>
</thead>
</table>

If $^{36}\text{Cl}$ ratio is much greater than 2000 $\times 10^{-15}$, the sample should be diluted with stable chloride carrier to reduce the ratio. If this step is necessary, enter the data below

Stable carrier solution Solution ID ________________, Solution Number ________________

Concentration ________________

Weight Carrier

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
<th>Sample 4</th>
<th>By</th>
</tr>
</thead>
</table>

Cl Concentration

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
<th>Sample 4</th>
<th>By</th>
</tr>
</thead>
</table>

Sample Preconcentration

NOTE: Fresh resin should be used for each sample to avoid the risk of cross contamination

Prepare an ion exchange chromatographic column, 10 mm ID by 200 mm length, containing a 5 cm bed of analytical grade 1-X8 anion exchange resin, Vendor/Lot ________________ ________________ By

Condition the column with 150 mL 1.5 M nitric acid solution (SW0008), Solution Number ________________. Test for chloride by adding 3 drops of 1 M silver nitrate solution to a suitable test tube, and collect the last 5 mL of eluant. If any turbidity is observed, condition the column with an additional 50 mL of the 1.5 M nitric acid and repeat test. Continue until test shows no turbidity. ____________________ By

NOTE: Discard test solutions in an appropriate silver waste container.
When chloride test is negative, wash column with 300 mL of 18 MΩ water, leaving 2 to 3 mL on the resin bed until ready to use. ____________________ By
Pour samples over column to concentrate the chloride on the column. Collect all eluant in a suitable container.

When all the sample aliquot is through the column, add 10 mL of 0.1 M ammonium hydroxide solution (SW0001), Solution Number ____________, to the column. Drain to the top of the resin.

Add 10 mL of 0.05 M nitric acid solution (SW0005), Solution Number ____________, to the column. Drain to the top of the resin.

Replace the catch container with a 30 mL centrifuge tube containing 10 drops of 1 M silver nitrate solution (SW0009), Solution Number ____________. Add 5 mL of 0.15 M nitric acid solution, Solution Number ____________,. Drain to the top of the resin.

Add 20 drops of low chloride, concentrated nitric acid, Vendor/Lot _________________. Cap tube, shake to coagulate silver chloride, and place in the refrigerator overnight Date ____________

NOTE: Discard contents of catch container if chloride has been removed from sample aliquot.

Note any comments or observations below (enter none if none):

Sample 1
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 2
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 3
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 4
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
Chloride Purification

NOTE: Fresh resin should be used for each sample to avoid the risk of cross contamination

Prepare an ion exchange chromatographic column, 10 mm ID by 200 mm length, containing a 5 cm bed of analytical grade 1-X8 anion exchange resin, Vendor/Lot ____________________________ By

Condition the column with 150 mL 1.5 M nitric acid solution (SW0008), Solution Number ____________. Test for chloride by adding 3 drops of 1 M silver nitrate solution to a suitable test tube, and collect the last 5 mL of eluant. If any turbidity is observed, condition the column with an additional 50 mL of the 1.5 M nitric acid and repeat test. Continue until test shows no turbidity. By

NOTE: Discard test solutions in an appropriate silver waste container.

When chloride test is negative, wash column with 300 mL of 18 MΩ water, leaving 2 to 3 mL on the resin bed until ready to use. By

Remove centrifuge tubes containing samples from the refrigerator, and centrifuge in an IEC Centra 4B centrifuge equipped with a type 224 rotor, 3224 cup, and 7231 cup adapter, at 100% power (about 2600 rpm), or equivalent, for 20 minutes By

Remove sample tubes from centrifuge and carefully remove supernatant with a plastic disposable transfer pipette and discard. By

NOTE: Discard supernatant in an appropriate silver waste container.

Add about 5 mL of 18 MΩ water and 20 drops of low chloride, concentrated ammonium hydroxide, Vendor/Lot ____________________________, to dissolve the silver chloride precipitate. Agitate with the pipette until completely dissolved. By

NOTE: Leach bottles in 3% nitric acid solution for at least 1 hour prior to using.

Drain wash to the top of the resin bed and place a 30 mL plastic bottle, (Nalge 2002-0001) under the column to collect the eluant. Load the dissolved ammonical silver chloride complex onto the column with the transfer pipette and drain to the top of the resin bed. By

Add 10 mL of 0.1 M ammonium hydroxide solution (SW0001), Solution Number ________________, to the centrifuge tube. Rinse tube and transfer to the column. Drain to the top of the resin. By

Add 10 mL of 0.05 M nitric acid solution (SW0005), Solution Number ________________, to the centrifuge tube. Rinse tube and transfer to the column. Drain to the top of the resin. By

Replace the catch container with a 30 mL centrifuge tube containing 10 drops of 1 M silver nitrate solution (SW0009), Solution Number ________________. Add 5 mL of 0.15 M nitric acid solution, Solution Number ________________. Drain to the top of the resin. By

Add 20 drops of low chloride, concentrated nitric acid, Vendor/Lot ____________________________. Cap tube, shake to coagulate silver chloride, and place in the refrigerator overnight.
NOTE: Discard contents of 30 mL bottle in an appropriate silver waste container

Note any comments or observations below (enter none if none):

Sample 1
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 2
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 3
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 4
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Final Target Preparation

Remove centrifuge tubes containing samples from the refrigerator, and centrifuge in an IEC Centra 4B centrifuge equipped with a type 224 rotor, 3224 cup, and 7231 cup adapter, at 100% power (about 2600 rpm), or equivalent, for 20 minutes

Date removed ________________    _______

By

Label and tare a 1.5 mL micro centrifuge tube (Fisher 05-407-10, or equivalent) for each sample. Enter tare weight as indicated below.

By

Carefully remove all the supernatant from the precipitate in the centrifuge with a transfer pipette. Carefully rinse precipitate with about 1 mL 18 MΩ water and discard. Add about 1 mL 18 MΩ water, and using the pipette, dislodge and draw up the precipitate. Transfer it to the micro centrifuge tube. Wash the tube with
about 1 mL 18 M$_2$O$_4$ water to collect any remaining precipitate and transfer it to the micro centrifuge tube.

Cap the micro centrifuge tube, place in a polycarbonate tube, and centrifuge as above for 20 minutes.

Remove the supernatant with the transfer pipette and place the micro centrifuge tube in a 60° C oven overnight.

When dry, remove the tube, cap, and re-weigh. Calculate AgCl weight, final - tare, and Cl weight (AgCl x 0.24735).

<table>
<thead>
<tr>
<th>Final Weight (g)</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
<th>Sample 4</th>
<th>By</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tare Weight (g)</td>
<td>Sample 1</td>
<td>Sample 2</td>
<td>Sample 3</td>
<td>Sample 4</td>
<td>By</td>
</tr>
<tr>
<td>AgCl Weight (mg)</td>
<td>Sample 1</td>
<td>Sample 2</td>
<td>Sample 3</td>
<td>Sample 4</td>
<td>By</td>
</tr>
<tr>
<td>Cl Weight (mg)</td>
<td>Sample 1</td>
<td>Sample 2</td>
<td>Sample 3</td>
<td>Sample 4</td>
<td>By</td>
</tr>
</tbody>
</table>

Note any comments or observations below (enter none if none):

Sample 1
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 2
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 3
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

Sample 4
_________________________________________________________________________________
Label microcentrifuge tube with PRIME LAB ID, isotope and replicate code, and user ID. Submit sample in an appropriate storage box for AMS measurement.

Date Completed _______________   _______  By

Date Submitted _______________   _______  By