

**PRIME Laboratory
Purdue University**

Chemistry Operations

Analytical Worksheet

TITLE : Isolation And Purification of Chloride from Low Level Water Samples for AMS,
 Method AW0004-003 Effective January 2, 2007

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Sample Identification and Information

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

Chemistry Project Number _____ Submitter _____

Isotopes of Interest Al ₃ Be ₁ Ca ₆ Cl X₅ I 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 4

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

 Sample 1

 Sample 2

 Sample 3

 Sample 4

 By

If ^{36}Cl ratio is much greater than 2000×10^{-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

 Sample 1

 Sample 2

 Sample 3

 Sample 4

 By

Cl Concentration

 Sample 1

 Sample 2

 Sample 3

 Sample 4

 By

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 M Ω 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.

_____ By

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.

_____ By

Add 10 mL of 0.05 M nitric acid solution (SW0005), Solution Number _____, 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

Date _____

_____ By

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

Date _____

By

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_ water to collect any remaining precipitate and transfer it to the micro centrifuge tube.

By

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

By

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

By

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

Final Weight (g)	_____	_____	_____	_____	_____
	Sample 1	Sample 2	Sample 3	Sample 4	By
Tare Weight (g)	_____	_____	_____	_____	_____
	Sample 1	Sample 2	Sample 3	Sample 4	By
AgCl Weight (mg)	_____	_____	_____	_____	_____
	Sample 1	Sample 2	Sample 3	Sample 4	By
Cl Weight (mg)	_____	_____	_____	_____	_____
	Sample 1	Sample 2	Sample 3	Sample 4	By

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