

# Measurement of $^{36}\text{Cl}$ and $^{129}\text{I}$ in Water

**Summary of Method** Chlorine-36 and Iodine-129 are separated and measured from up to 500mL aliquots of water. Samples are adjusted to 0.1-1.0M  $\text{H}_2\text{SO}_4$  and 0.1M  $\text{SnSO}_4$  is added to ensure reduction of any oxidized species to chloride ( $\text{Cl}^-$ ) and Iodide ( $\text{I}^-$ ). The CL resin is prepared by loading with  $\text{Ag}^+$  cations which will facilitate the retention of  $\text{Cl}^-$  and  $\text{I}^-$ . The sample is loaded onto the CL Resin, the column is rinsed with deionized water, and  $\text{Cl}^-$  is recovered using 0.1M ammonium thiocyanate ( $\text{NH}_4\text{SCN}$ ). If iodine determination is also required, the resin is the rinsed with 0.1%  $\text{NaOH}$  and then  $\text{I}^-$  is recovered using 0.35M sodium sulfide ( $\text{Na}_2\text{S}$ ).  $^{36}\text{Cl}$  ( $t_{1/2} = 3.01\text{E}5$  years,  $\beta^-_{\text{mean}} = 251\text{keV}$ ,  $\beta^-_{\text{max}} = 709.55\text{keV}$ , abundance = 98.1% ) and  $^{129}\text{I}$  ( $t_{1/2} = 1.57\text{E}7$  years,  $\beta^-_{\text{mean}} = 40.03\text{keV}$ ,  $\beta^-_{\text{max}} = 149.3\text{keV}$ , abundance = 100% ) can then be measured using liquid scintillation counting.

## Reagents

Cl Resin Bulk (CL-B50-A or CL-B50-S)\*

-or-

Cl Resin Prepacked 2mL columns (CL-C50-A)

Deionized Water

$\text{NaOH}$

$\text{H}_2\text{SO}_4$

$\text{SnSO}_4$

$\text{Na}_2\text{S}$

$\text{NH}_4\text{SCN}$

$\text{AgNO}_3$  (or Solution of 10mg/mL  $\text{Ag}^+$ )

Liquid Scintillation Cocktail

\*If packing own columns for gravity flow, then use CL-B50-A. If using vacuum assisted flow, then use CL-B50-S.

## Equipment

Centrifuge tubes - 50mL

Liquid scintillation vials, 20mL glass

Liquid scintillation counter

Calibrated pipets and disposable tips

Appropriately sized glass beakers and flasks

Analytical balance

Filter funnel and paper

**Optional:** Empty columns and frits for packing own columns

## Sample Preparation

Up to 500mL of water sample in glass beaker.

Adjust to 0.1-1.0M  $\text{H}_2\text{SO}_4$ .

Add 1 mL of 0.1M  $\text{SnSO}_4$  per 50mL of sample.

Mix sample well.

## CL Resin Preparation (Batch Mode)

**Reagent amounts may be adjusted proportionally to as needed.**

Weigh 10 grams CL Resin into a 250mL flask.

Dissolve 0.65 grams  $\text{AgNO}_3$  in 100mL 1M  $\text{H}_2\text{SO}_4$ .

Add  $\text{Ag}/\text{H}_2\text{SO}_4$  solution to resin and Mix for 2 hrs.

Filter CL Resin and rinse twice with 1M  $\text{H}_2\text{SO}_4$ .

Slurry resin in 100mL 0.1M  $\text{H}_2\text{SO}_4$  and pack into the appropriate sized column.

## CL Resin Preparation (Column)

**Reagent amounts may be adjusted**

Slurry resin in 100mL 0.1M  $\text{H}_2\text{SO}_4$  and pack into the appropriate sized column or obtain prepacked 2mL column of CL Resin.

Rinse 2mL column with 10mL 1M  $\text{H}_2\text{SO}_4$ .

Dissolve 0.65 grams  $\text{AgNO}_3$  in 100mL 1M  $\text{H}_2\text{SO}_4$ .

Load 2 mL of solution onto each 2mL column.

Wait 2hr for  $\text{Ag}$  to be completely absorbed.

Rinse 2mL column with 10mL 1M  $\text{H}_2\text{SO}_4$ .

## Chlorine/Iodine Separation

- 1) Load Sample onto 2mL CL Resin column at 2mL/min.
- 2) Rinse 2mL CL Resin column with 10mL 0.1M H<sub>2</sub>SO<sub>4</sub>.
- 3) Rinse 2mL CL Resin column with 10mL deionized water.
- 4) Strip 2mL CL Resin into 20mL LSC vial with 5mL 0.1M NH<sub>4</sub>SCN to recover chloride.
- 5) Rinse 2mL CL Resin column with 10mL 0.1% NaOH.
- 6) Strip 2mL CL Resin column into a 2mL LSC vial with 5mL 0.35M Na<sub>2</sub>S.
- 7) Add 15 LSC cocktail to each LSC vial.
- 8) Count <sup>36</sup>Cl and <sup>129</sup>I samples by LSC.

Decontamination Factors (DF) for Chloride and Iodide Fractions		
Analyte	DF Cl- fraction	DF I- fraction
Ba	>1000	>600
Cd	>6900	>7700
Cu	>210	>190
Mn	>210	>370
Ni	>170	>320
Pb	>300	>720
U	>1900	>200
<sup>60</sup> Co	>320	>320
<sup>137</sup> Cs	>150	>150
<sup>90</sup> Sr/ <sup>90</sup> Y	>180	>160
<sup>36</sup> Cl	N/A	>160
<sup>129</sup> I	>420	N/A

Retention of Cl and I on CL Resin from 1M H <sub>2</sub> SO <sub>4</sub>	
Analyte	Dw
<sup>36</sup> Cl	1600
<sup>129</sup> I	1980

Chloride capacity: 4 mg / 2mL column  
Iodide capacity: 15 mg / 2mL column

## References

- 1) A. Zulauf, S. Happel, M.B. Mokili, A. Bombard, H. Jungclas, "Characterization of an extraction chromatographic resin for the separation and determination of <sup>36</sup>Cl and <sup>129</sup>I." J. Radioanal. Nucl. Chem. 286(2), 539-546 (2010).