1 Apparatus

1.1 *Analytical balance- 0.0001 g sensitivity*

1.2 *Beaker (10 mL, 50 mL)*

1.3 *Vortex mixer*

1.4 *20 mL PE vials*

1.5 *Pipettes*

1.6 *Fume hood*

1.7 *Hotplate*

1.8 *Column reservoirs - 25 mL, Part number AC-120*

1.9 *Column rack - Part number AC-104*

Alternatively:

1.10 *Vacuum box or vacuum bottle with column adaptor*

1.11 *Vacuum pump*

2 Reagents

2.1 *All references to water should be understood to mean deionized water.*

2.2 *Bulk CL resin or CL resin columns –CL resin supplied **NOT** loaded with* Ag^{+}

2.3 *1M sulfuric acid*

2.4 *Optional: 0.1M* $SnSO_4$ *solution*

2.5 *Ag load solution (20* $mg.mL^{-1}$*) - Dissolve 787,4 mg* $AgNO_3$ *in 25 mL water*

2.6 *0.1M* NH_4SCN *solution (alternatively 0.1M NaSCN might be used)*

2.7 *1% (w/w) NaOH solution*

2.8 *0.35M* Na_2S *solution*

Note 1: all work with the Na_2S should be performed in a fume hood, including the addition of the liquid scintillation cocktail.

2.9 *Liquid scintillation cocktail*

Note 2: Some LSC cocktails may reduce traces of Ag^+ co-eluted from the column resulting in 'blackened' LSC samples, it is thus advisable to test your cocktail before use. ProSafe HC was found to be suited.

3 Procedure

3.1 *Column preparation (alternatively use pre-packed CL columns):*

3.1.1 Weigh 0.65g of the resin into a 20 mL PE vial

3.1.2 Add 5 mL of 1M sulfuric acid and soak for at least 30 min, preferably while shaking

3.1.3 Pack 2 mL standard column with the soaked resin

3.1.4 Add 1 mL of Ag^+ load solution onto the column. Allow the solution to load onto the column, then close the lower end of the column to stop the flow. Let the column stand for more than 30 min in order to allow for Ag uptake.

Note 3: Alternatively the Cl resin might also be loaded with Ag^+ in batch mode; in this case perform steps 3.1.1 and 3.1.2, then add 1 mL Ag^+ load solution into the PE vial, cap and shake for another 30 min (preferably longer). Transfer the Ag^+ loaded resin into a 2 mL standard column.

3.1.5 Add 5 mL 1M H_2SO_4 onto the column and open the lower end of the column. Allow the solution to pass

Note 4: In case the sample is loaded in 6 mM Na_2CO_3 soak resin in water and rinse column with water instead of 1M H_2SO_4; rinse until the eluate is free of acid.

3.2 *Sample preparation:*

3.2.1 Water samples: evaporate sample to a volume of approx. 10 mL, adjust to 1M sulfuric acid.

Note 5: 1M sulfuric acid is chosen as Ag^+ retention is optimal under these conditions, near neutral pH is also acceptable.

3.2.2 Bubbler solutions:

3.2.2.1 0.1M and 1M NaOH solutions should be at least neutralized using sulfuric acid, preferably the loading solutions should be adjusted to 1M H_2SO_4.

Note 6: in case only I-129 is to be analysed the NaOH solutions can be used as they are without adjustment.

3.2.2.2 6 mM Na_2CO_3 solutions can be employed as they are, no adjustment is necessary.

3.2.3 Solid residues: Dissolve residue in 5 mL 1M sulfuric acid.

Note 7: The concentration of natural Cl^- and I^- should preferably be determined in the sample load solution e.g. by ion chromatography.

Note 8: In order to assure that Cl and I are present in the sample as chloride and iodide it is advisable to additionally add a reducing agent (e.g. 0.1M $SnSO_4$) to the sample loading solution.

3.3 *Cl/I separation:*

3.3.1 Load sample onto column and allow to drain.

3.3.2 Rinse beaker with 5 mL 1M sulfuric acid (optional: deionized water) and add onto the column.

Note 9: In case the sample was loaded in 6 mM Na_2CO_3 deionized water has to be used instead of 1M H_2SO_4.

3.3.3 Rinse column with 2 x 5 mL deionised water. Discard eluates.

Note 10: In case the sample was loaded in 6 mM Na_2CO_3 the column should be rinsed with 5 mL 0.1M H_2SO_4 between both water rinsing steps in order to improve C-14 removal.

3.3.4 Place fresh 20 mL PE vial below column.

3.3.5 Elute Cl^- with 5 mL 0.1M NH_4SCN.

Optional: take aliquot for Cl content determination in order to allow for yield determination.

3.3.6 Add 10 mL LSC cocktail and shake vigorously.

3.3.7 Replace LSC vial by waste container.

3.3.8 Rinse column with 10 mL 1% NaOH.

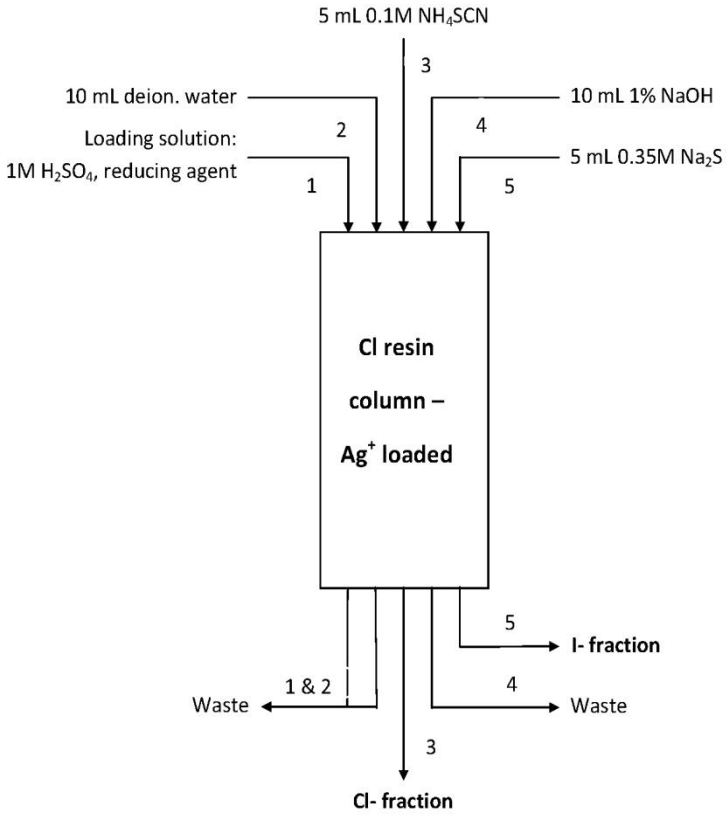
3.3.9 Place fresh 20 mL PE vial below column.

3.3.10 Elute I^- with 5 mL 0.35M Na_2S.

Optional: take aliquot for I content determination in order to allow for yield determination.

3.3.11 Add 10 mL LSC cocktail and shake vigorously.

3.4 *Synopsis of the separation*



4 References

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