RiO5 METHOD (46)

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Naturally occurring ^{32}**P** and ^{33}**P** measurements via Liquid Scintillation Counting

Fresh and salt water matrices

Disclaimer

It is the responsibility of the analyst to follow established safety and health practices. Although each laboratory identified as the source has tested the methods, each user should perform an individual validation procedure.

Table of Contents

1 SCOPE

This procedure provides basic information regarding the collection and purification of environmental samples for the measurement of naturally occurring ^{32}P and ^{33}P via liquid scintillation counting.

2 EQUIPMENT and CHEMICAL REAGENTS

Specific equipment and consumables used are listed below. Note that not all items are currently available and may need to be sourced or new equipment or consumables tested.

2.1 Equipment and consumables

Seawater sample collection

- 1 µm polypropylene Hytrex™ prefilters, and two 0.2 µm pleated polypropylene membrane cartridges (25.4 cm long, 7.6 cm diameter)
- Three parallel 61 cm long, 7.6 cm diameter PVC pipes (volume 2.7 L)
- 25 µm pore size polypropylene sheets (MWM Co., 1 Newbury St., Quincy, MA 02171)
- 0.75 horsepower bronze gear pump (Teel, 1B416)
- Mn impregnated cartridges (see ^{234}Th procedure)
- 934 AH GF/F Filters
- AG1 x 8 100-200 mesh resin (Bio-Rad)
- Amberlite IRC-718 (iminodiacetate group, Rohm and Haas Co.)
- 17 mL nonstatic liquid scintillation (LS) vial (available from Packard Industries)
- Hot Plates
- Combustion Oven
- Packard Tri-Carb 2750TR/LL LSS (Packard Instrument Co.).

2.2 Tracers

- Artificially produced ^{32}P and ^{33}P used to calibrate recovery and instrument.

2.3 Chemical Reagents & Solutions

- 6.25 N NaOH (Certified ACS)
- 50% FeCl3 solution (Certified ACS) purified with di-isopropyl ether to minimize stable P contamination.
- 8 N HNO3 (ACS Certifed)
- 8N HCl (ACS Certifed)

- 30% H202 (ACS Certified)
- Concentrated NH4OH (ACS Certified)
- Amonium Molybdate Solution (60 g MoO3 + 440 mL H2O + 60 ml NH4OH poured into 500mL of 50% HNO3)
- MgCl2/NH4Cl2 Solution (55 gMgCl2 + 70 g NH4C1 + 300mL conc. NH4OH diluted to 1 L and filtered)
- Ultima Gold AB (Packard) scintillation cocktail

3 PROCEDURE

^{32}P/^{33}P Sample Collection: Seawater dissolved and particulate samples

1. Pack 3 L pipe with 90 purified $Fe(OH)_3$ filters. Attach flow meter to outlet
2. From ship's seawater line: Pass water sequentially from highest to lowest pore size pre-filters (i.e. through 10 μm, then 1 μm, then 0.2 μm), followed by Mn Carts, and then into $Fe(OH)_3$ pipe.
3. Use ½ inch tubing and hose clamps for setup.
4. Turn on seawater flow such that flow rate is between 1.2 and 1.4 g/min (use flow meter to estimate rate).
5. Record start time and start volume.
6. Collect water from inlet at start and every 5 hours (Start of day, Middle of day, and End of Day). Make sure to Mark the sample. Collect water from outlet (filtrate) every hour. Use acid-cleaned polypropylene 125 mL plastic bottles and store in refrigerator after collection. Record time and volume.
7. Try to filter > 5000 L.
8. Filtration does not need to be continuous. May start and stop at beginning/end of day.

$^{32}P/^{33}P$ Log Sheet

Cruise: _____

Date: _____

#Fe filters: _____

Start Volume: _____ **End Volume: _____**

Start Date/Time: _____ **End Date/Time: _____**

Prefilters Used: _____

Mn Carts Used?: _____

Mark Samples collected before Fe Pipe with *

Sample No:	Time Coll.	Volume coll.	Sample No:	Time Coll.	Volume coll.

$^{32}P/^{33}P$ Sample Purification

Details regarding sample purification are provided in Benitez-Nelson and Buesseler (1998) (See Chemical Purification Flow Chart below). Below is a checklist to help guide users through the sample purification steps.

32P/33P Analysis for Seawater Sample Checklist

Cruise:

Date:

Sample Type:

Purification Step		**Sample**	**Sample**	**Sample**	**Sample**
Samples Ashed?					
Acid Extraction					
Ether Extraction					
Filtration					
Recovery sample taken (5 mL to 50mL)					
Volume:					
Ammonium Molybdate ppt. (volume added?)					
Magnesium Ammonium ppt. (volume added?)					
10 mL AG1 x 8 column					
	50 mL precondition				
	Sample				
	3 x 2 mL rinses				
	20 mL rinse				
Evaporate and Redissolve					
10 mL IRC 718 column					

Purification Step		Sample	Sample	Sample	Sample
	50 mL precondition				
	Sample				
	3 x 2 mL rinses				
	20 mL rinse				
Evaporate and transfer to LS Vial					
LS Vial Tare					
LS Vial Total					
Recovery sample taken (50 µL to 10mL)					

4 REFERENCES

Benitez-Nelson, C.R. and K. O. Buesseler (1998) New techniques for the measurement of ^{32}P and ^{33}P activities in rain and seawater. *Anal. Chem.*, **70**, 64-72.

Benitez-Nelson, C.R. and K. O. Buesseler (1999) Temporal variability of inorganic and organic phosphorus turnover rates in the coastal ocean. *Nature*, **398**, 502-505.

Benitez-Nelson, C. R. and D. M. Karl (2002) Phosphorus cycling in the North Pacific Subtropical Gyre using cosmogenic ^{32}P and ^{33}P. *Limnol. and Ocean.*, **47**, 762-770.

5 FLOW CHART

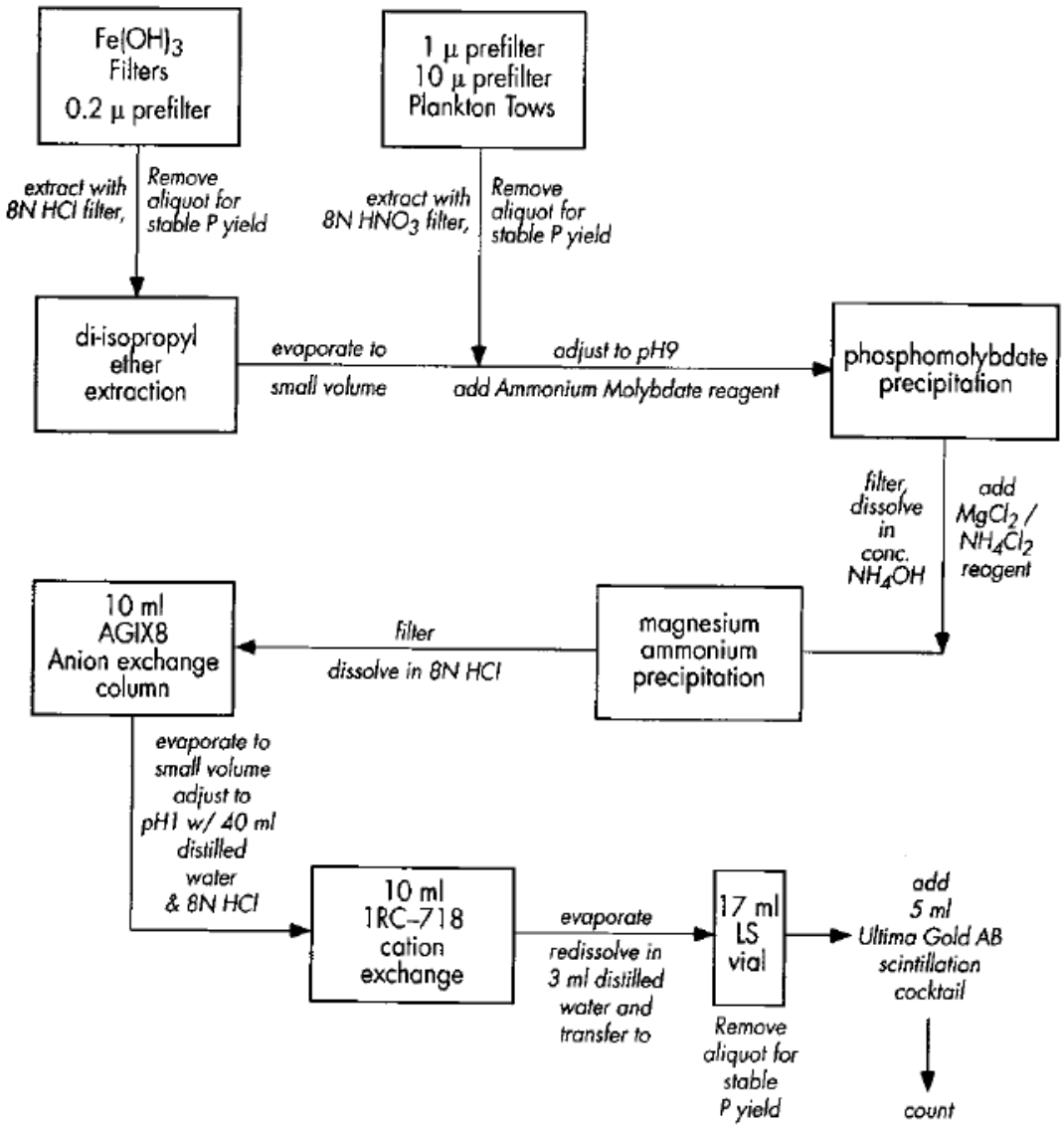

Figure 2. Chemical purification scheme.

From Benitez-Nelson and Buesseler (1998)