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Comment on “How accurate are ^{234}Th measurements in seawater based on the MnO_2 -impregnated cartridge technique?” by Pinghe Cai et al.

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1. Introduction

[1] The paper by Cai *et al.* [2006] poses the question whether the use of MnO_2 -impregnated cartridges can provide accurate measurements of dissolved ^{234}Th concentrations in seawater. Cai *et al.* [2006] compared the MnO_2 -impregnated cartridge technique with the small-volume MnO_2 coprecipitation technique in eight samples taken in

the South China Sea. On average, $\sim 30\%$ of the dissolved ^{234}Th appeared to be missing from their MnO_2 cartridges, and this apparent loss they attributed to organic complexation of dissolved ^{234}Th in seawater. If true, this calls into question previous measurements of ^{234}Th made using MnO_2 -cartridges [e.g., Livingston and Cochran, 1987; Buesseler *et al.*, 1992; Cochran *et al.*, 1995; Moran *et al.*, 1997; Baskaran *et al.*, 1993, 1996; Santschi *et al.*, 1999;

Table 1. Results of Extraction Efficiency Determinations for Dissolved ^{234}Th Using MnO_2 Impregnated Filters in Seawater From Various Investigators^a

Reference	[^{234}Th] in Deep Water, dpm/L	Location	Flow Rate, L/min	Cartridge Type	Number (n) of Samples	Mean Extraction Efficiency (equation (1)), %
<i>Bacon et al.</i> [1996]	2.4	equatorial Pacific	1–8.5	0.5 μm^{b}	120	90.5 ± 12.7
<i>Baskaran et al.</i> [1993]	2.4	Gulf of Mexico	25–35	0.5 μm^{c}	20	93 ± 5
<i>Baskaran et al.</i> [1996]	2.4–2.5	Gulf of Mexico	20–30	0.5 μm^{c}	NR	NR
<i>Buesseler et al.</i> [1992]	2.4–2.5	JGOFS NABE	4–8	1 μm^{b}	40	87 ± 8
<i>Cai et al.</i> [2006]	2.5	South China Sea	4–7	5 μm^{d}	9	61 ± 53 ^e
<i>Cochran et al.</i> [1995]	-	Arctic	5–16	0.5 μm^{b}	83	50–95
<i>Hartman and Buesseler</i> [1994]	-	equatorial Pacific	4	5 μm^{d}	104	79 ± 8
<i>Hung et al.</i> [2004]	2.4	Gulf of Mexico	10–20	0.5 μm^{f}	NR	NR
<i>Guo et al.</i> [2002]	2.4	Gulf of Mexico	10–20	0.5 μm^{f}	NR	NR
<i>Livingston and Cochran</i> [1987]	2.4	North Atlantic	2–7	1 μm^{b}	12	83 ± 8
<i>Moran et al.</i> [1997]	2.4	Arctic Ocean	2–3	5 μm^{d}	30	90 ± 7
<i>Santschi et al.</i> [1999]	2.5–2.9	North Atlantic; Lab	10–35; 35	0.5 μm^{f}	18; 2	86 ± 9 ^g ; 99 ± 1
L. Guo et al., unpublished data, 2007	1.9–2.0	northern Gulf of Mexico	10	0.5 μm^{f}	6	96.5 ± 1.5

^aNR, not reported.

^bCUNO Micro-WyndII Polypropylene, 25 cm.

^cPerforated Polypropylene (United Filters, Inc., Uoo.5R10P), 25 cm.

^dHytex II, 25 cm.

^eRecalculated from *Cai et al.* [2006], including the reported negative value.

^fSparkling Clear Polypropylene, 25 cm.

^gUnpublished notebook data.

Guo et al., 2002; *Hung et al.*, 2004]. However, previous applications of the MnO_2 -cartridge method have found it to give good agreement with ^{238}U in deep water and to agree with ^{234}Th measured on small volume samples. Moreover, the analytical procedures applied by *Cai et al.* [2006] are not typical of other workers. Here we show that the two methods yield comparable results, within errors, when properly applied.

2. Methodology

[2] The MnO_2 -cartridge method was developed initially for extraction of anthropogenic radionuclides (Pu isotopes and ^{241}Am) from large volumes of seawater by in situ pumping [*Winget et al.*, 1982; *Mann et al.*, 1984] and adapted by *Livingston and Cochran* [1987] and *Cochran et al.* [1987] to also extract Th isotopes (Th-232, Th-230, Th-228). This sampling technique had the advantage of collecting both particulate and dissolved radionuclides. The method was subsequently used for measurement of dissolved and particulate ^{234}Th and has been applied by many groups. Several different types, lengths and pore sizes of cartridges have been used (Table 1). Cartridges are generally wound fiber polypropylene with pore sizes of 0.5 μm [e.g., *Buesseler et al.*,

1992; *Cochran et al.*, 1993, 1995; *Bacon et al.*, 1996; *Baskaran et al.*, 1993, 1996; *Santschi et al.*, 1999; *Guo et al.*, 2002; *Hung et al.*, 2004], 1 μm [*Livingston and Cochran*, 1987; *Cochran et al.*, 1987; *Colley and Thomson*, 1994] or 5 μm [*Hartman and Buesseler*, 1994; *Moran et al.*, 1997]. The cartridges are usually preceded by prefilters that are either untreated cartridges or a series of filters of different pore sizes (e.g., Nitex or Teflon, Whatman GF/F or QM-A) that separate particulate Th. Thorium retained on the MnO_2 cartridges is assumed to be “dissolved.” The extraction efficiency of dissolved Th is calculated from the activities on two cartridges in series (A and B [*Livingston and Cochran*, 1987]):

$$\text{Efficiency}(\%) = 100(1 - B/A). \quad (1)$$

Dissolved Th is calculated from the activity on the A and B cartridges, decay-corrected to collection. Different flow rates and pumping systems have been used, but efficiencies are generally greater than 70%. *Cai et al.* [2006] used cartridges of 5 μm pore size and 5-inch (12.7 cm) length, while the pore size and cartridge length used by other groups are often very different (e.g., 0.5 μm , lengths from 8.3 to 25 cm; Table 1). Because adsorption of dissolved ^{234}Th is related to the contact area of MnO_2 , one could assume that larger pore sizes and

shorter cartridges would yield lower efficiencies at equal flow rates, a trend that is certainly evident from Table 1. As well, dependence of extraction efficiency on flow rate through the cartridge series has been observed [Cochran *et al.*, 1995; Charette and Moran, 1999]. Cai *et al.* [2006] reported “theoretical collection efficiencies” (equation (1)) of -70 to 95% (mean $61 \pm 53\%$). When used to calculate dissolved ^{234}Th from the MnO_2 cartridges, Cai *et al.* [2006] noted an offset relative to dissolved values derived from small volume samples (total Th minus particulate Th) and concluded that Mn cartridges were undercollecting dissolved Th.

[3] Any method that involves determination of radioactivity requires rigorous calibration and evaluation of standards and any analytical artifacts that might occur. The method used by most investigators for analysis of ^{234}Th on MnO_2 cartridges is nondestructive gamma spectrometry, applied to the ash remaining after igniting the cartridge in a furnace or to “pucks” produced by melting or compressing the cartridge. The method is standardized by adding a known amount of ^{234}Th in equilibrium with ^{238}U to a cartridge and counting it in the same geometry as the samples. This procedure works best with ashed samples because ashing more reliably produces a standard in which the ^{234}Th is homogeneously distributed. Contrary to the assertion of Cai *et al.* [2006], “many researchers” (Buessler *et al.* [1992] and Moran *et al.* [1997] are cited) do not “use deep water to calibrate their gamma detectors.” Instead, a ^{238}U standard (or other U-containing standard such as pitchblende) is used and deep water analyses are used as an independent check on the standardization (see example below).

[4] In contrast to the gamma spectrometric analysis of ashed MnO_2 cartridges, Cai *et al.* [2006] used a leaching procedure (2M HNO_3 , $30\% \text{H}_2\text{O}_2$) to remove the Th from the intact cartridge. The leachate was subsequently neutralized and Th was precipitated with $\text{Fe}(\text{OH})_3$. Yield tracers were added and yields were determined via alpha spectrometry. This procedure entails considerably more manipulation of the sample than does gamma spectrometry of cartridge ash. The few other cases in which a wet chemical procedure has been applied used mixtures of $\text{HCl}/\text{HNO}_3/\text{H}_2\text{O}_2$ [Colley and Thomson, 1994], $\text{HNO}_3/\text{HF}/\text{NH}_2\text{OH}\cdot\text{HCl}$ [Rutgers van der Loeff and Berger, 1993] and concentrated $\text{HCl}/\text{NH}_2\text{OH}\cdot\text{HCl}$ [Shimmield *et al.*, 1995]. Rutgers van der Loeff *et al.* [2006] cite a Soxhlet-type leaching technique using 6N HCl , requiring several days to produce a Th

recovery of $\sim 95\%$ from the cartridge. Wet chemical methods require equilibration of the tracer with the ^{234}Th on the cartridge, accurate calibration of all tracers and standardization of both alpha and beta counting systems.

[5] Cai *et al.* [2006] used the “dissolved” activity determined from small volume samples (total – particulate Th) to independently determine “derived collection efficiencies” for the A and B cartridges separately and found lower values than obtained from equation (1). However, such a discrepancy was not observed by others who used the same approach [e.g., Baskaran *et al.*, 1993, 1996; Santschi *et al.*, 1999]. For example, Santschi *et al.* [1999] found that field cartridge efficiencies were compatible with separate laboratory tests of MnO_2 cartridges using ^{234}Th added to 200 L that gave 99% cartridge extraction efficiency (Table 1).

[6] In our experience, MnO_2 -impregnated cartridges made by different individuals can vary in quality. As a consequence, QA/QC procedures when preparing MnO_2 cartridges for field sampling are essential, requiring laboratory calibration data to test that the MnO_2 -impregnated cartridges quantitatively extract ^{234}Th from seawater. However, Cai *et al.* [2006] did not provide such data.

3. Field Data: Method Verification and Intercomparison

[7] Our laboratories have applied the MnO_2 -cartridge method for over 20 years. Initial efforts to verify the accuracy of the technique involved comparison of the values obtained in deep water with ^{238}U activities. Because the cartridge method was independently calibrated in the laboratory, this comparison is a robust means of verifying the accuracy of the method. One such verification was undertaken in 1992–1993 as part of the JGOFS EqPac study (cartridge efficiency of $91 \pm 13\%$ [Bacon *et al.*, 1996]). Figure 1 shows 42 analyses of total ^{234}Th in deep water (depths $>500 \text{ m}$; all MnO_2 cartridges ashed) of the equatorial Pacific. This location, although subject to upwelling, is far from the influences of ocean margins that might affect $^{234}\text{Th}/^{238}\text{U}$ equilibrium. The results show that ^{234}Th is in equilibrium with ^{238}U (Figure 1). The standard deviation of the mean ^{234}Th activity is $\sim 8\%$, comparable to the 1σ error on an individual measurement.

[8] More recently, laboratories measuring ^{234}Th have begun using a small volume method that offers

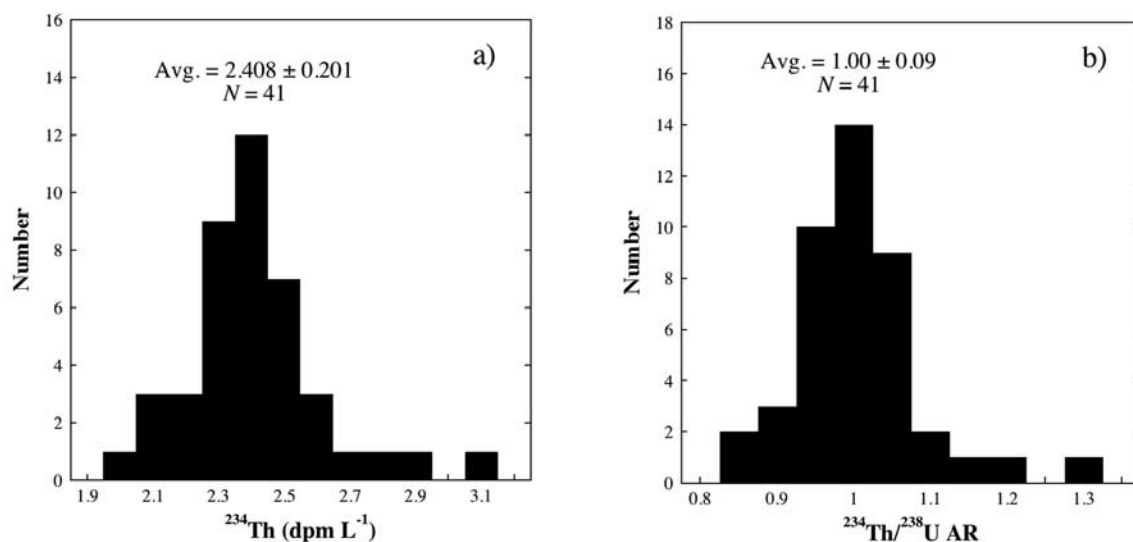


Figure 1. Histogram of (a) ^{234}Th activity and (b) $^{234}\text{Th}/^{238}\text{U}$ activity ratios (AR) in deep-water (>500 m) samples taken in the equatorial Pacific (1992–1993). Samples were collected by in situ pumping, using a series of 53 μm Nitex mesh, 1 μm Whatman QM-A, 0.7 μm Whatman GF/F and two CUNO MnO_2 cartridges. Cartridges were ashed, and ^{234}Th was determined by gamma spectrometry (K. Cochran, unpublished data, 2007, and Bacon *et al.* [1996]). The ^{238}U activity of the water is 2.40 dpm/L [Chen *et al.*, 1986].

increased sampling resolution but provides only total ^{234}Th . Comparison of total ^{234}Th derived from in situ pumping (gamma spectrometry; MnO_2 cartridges ashed) with total ^{234}Th (beta counting) in small volume samples from diverse locations shows no clear bias toward lower activities on MnO_2 cartridge-derived ^{234}Th (Figure 2a). A histogram treatment of the data shows that the mean ratio of small volume ^{234}Th to MnO_2 -derived ^{234}Th is 1.11 ± 0.23 (Figure 2b). Figure 2b includes many samples from the upper water column, where $^{234}\text{Th}/^{238}\text{U}$ disequilibrium is most pronounced. For these samples, some of the differences between the methods may be real, caused by short-term temporal variations of ^{234}Th in the upper water column [Charette *et al.*, 1999]. The data of Cai *et al.* [2006] stand out in Figure 2a as among the most discrepant of the data and are clearly distinct from large numbers of other data (Figure 2b versus Figure 2c).

[9] Cai *et al.* [2006] implied that the ^{234}Th deficit in nepheloid layers from continental slope areas as documented, for example, by Santschi *et al.* [1999] and Baskaran *et al.* [1996], was an artifact of the MnO_2 -impregnated cartridge method. However, Santschi *et al.* [1999] documented high cartridge extraction efficiencies from both laboratory (99%, Table 1) and field ($86 \pm 9\%$, Table 1) data, and equilibrium values of $^{234}\text{Th}/^{238}\text{U}$ ratios were noted at depths where aggregate concentrations were lowest. Indeed the observed deficits coincid-

ed with increased abundances of aggregate numbers in nepheloid layers. Furthermore, deficits in $^{234}\text{Th}/^{238}\text{U}$ disequilibria in deep waters have also been reported for other oceanic environments such as bottom nepheloid layers and ocean margins (e.g., Bacon and Rutgers van der Loeff [1989] for the Pacific, and Moran and Smith [2000] for the Arctic), and these documented deficits cannot be taken as a “proof” that the MnO_2 -cartridge method is invalid. Finally, Hung and Gong [2007], using large-volume 0.5 μm MnO_2 cartridge extraction methods (D of Table 1), found ^{234}Th to be in equilibrium with ^{238}U at 140 m in a nutrient-depleted region close to where Cai *et al.* [2006] reported their profile.

4. Summary

[10] Our collective experience is that the preferred method for treating MnO_2 cartridges is ashing followed by nondestructive gamma spectrometry of the ash. This approach facilitates the independent preparation of standards and allows deep water to be used as a check on accuracy of the method (e.g., Figure 1). Such an approach provides a clear answer to the question “How accurate are ^{234}Th measurements in seawater based on the MnO_2 -impregnated cartridge technique?” In contrast to the assertions of Cai *et al.* [2006] based on eight samples, the large number of MnO_2 cartridge samples analyzed over the past 20 years shows that

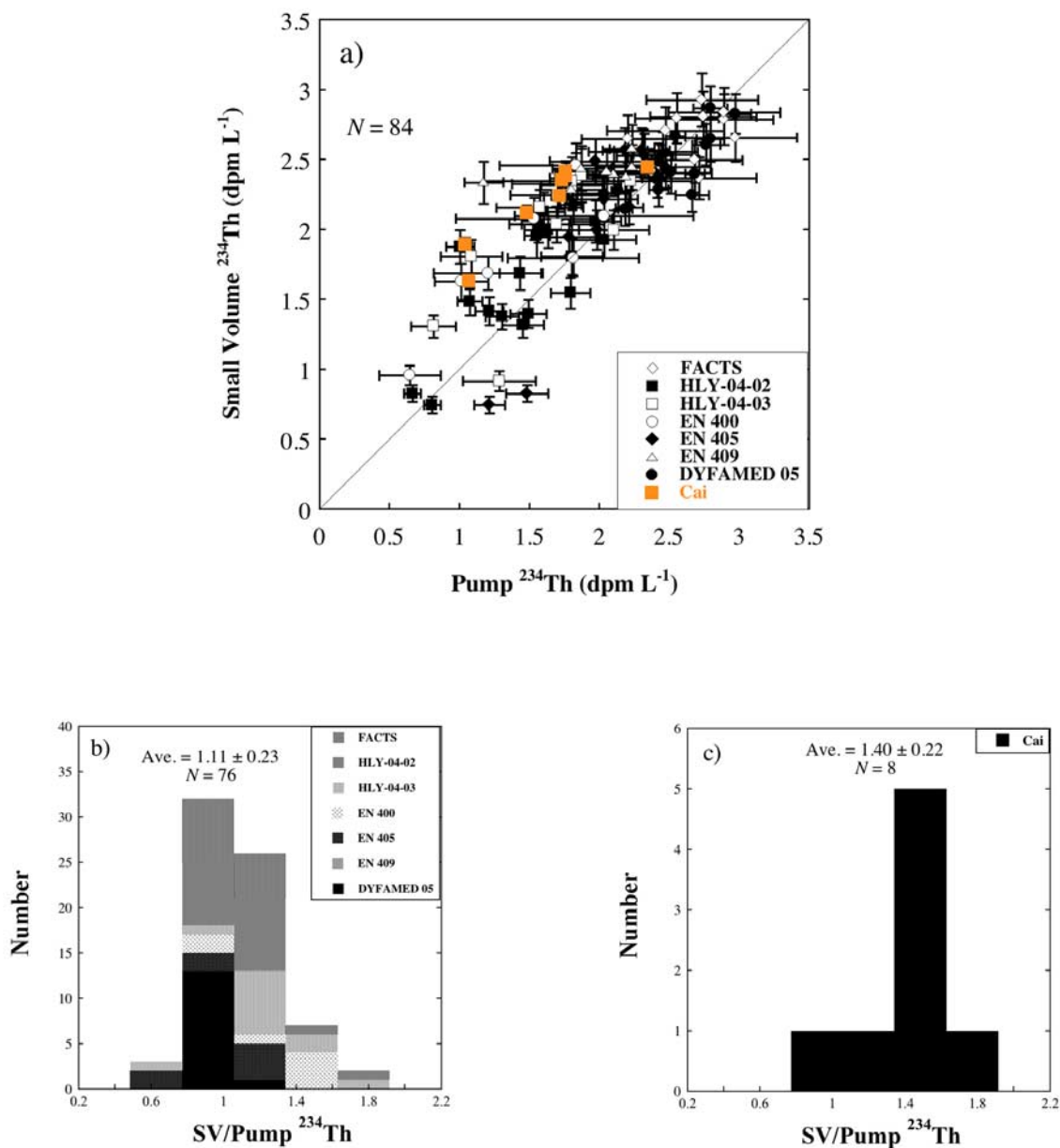


Figure 2. (a) Plot of ^{234}Th activity determined by small volume (SV ^{234}Th) versus in situ pump with MnO_2 cartridges (Pump ^{234}Th) based on published or submitted data from: south Aegean Sea (FACTS [Speicher *et al.*, 2006]); Chukchi Sea, western Arctic Ocean (HLY 04-02, HLY 04-03 [Lepore *et al.*, 2007]); western Mediterranean Sea (EN 405 [Lepore *et al.*, 2007]); northwest Atlantic, between RI and Bermuda (EN 400, 409; K. Lepore *et al.*, Testing the *p*-ratio in the Mediterranean Sea and NW Atlantic Ocean: Implications for the accuracy of ^{234}Th -derived POC export flux, unpublished manuscript, 2007); Mediterranean Sea, off Monaco (DYFAMED (K. Cochran *et al.*, Time-series measurements of ^{234}Th in water column and sediment trap samples from the northwestern Mediterranean Sea, submitted to *Deep Sea Research, Part II*, 2007)); South China Sea [Cai *et al.*, 2006]. (b) Histogram of SV/Pump ^{234}Th ratios indicating $11 \pm 22\%$ higher SV ^{234}Th activities for field studies listed ($N = 76$). (c) Histogram of SV/Pump ^{234}Th ratios indicating $40 \pm 22\%$ higher SV ^{234}Th activities based on a total eight samples reported in the South China Sea by Cai *et al.* [2006].

the method provides acceptable accuracy, with precision on the order of 10%, and is in good agreement with total ^{234}Th determined on small volume samples. However, there is clearly a need

for future research on the speciation of Th in seawater. Organic complexation may well be important in coastal or freshwater settings and may affect the geochemical behavior of Th. In open

ocean settings, our results show that the extent of complexation, at least as reflected in MnO₂ cartridge measurements of ²³⁴Th, cannot be as great as Cai *et al.* [2006] suggest.

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