

TRACE ORGANIC SAMPLER INTERCALIBRATION RESULTS

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SUMMARY

This report compares the levels of organochlorine and polynuclear aromatic compounds in water from the San Francisco Bay generated during the June 1996 and January 1997 sampler intercalibration exercise. In this intercalibration, two trace organic sampling systems were compared side-by-side in six locations (1996-Redwood creek BA40, Coyote creek BA10, and Golden Gate BC20; 1997-Sacramento River BG-20, San Joaquin River BG-30, and Standish Dam BW-10). The two sampling systems compared were a polyurethane foam (for collection of the dissolved phase)- glass fiber filter (particulate) and an XAD column (dissolved) -fiber glass cartridge system (particulate). In addition, for comparison, data generated during previous RMP cruises are compared to the intercalibration results to determine the magnitude of temporal variation.

In general, levels of pesticides, PCBs, and PAHs are similar between the XAD sampler and BBI sampler. In addition, comparison of temporal trends in past RMPs show the data generated by the XAD sampler to be very similar to past RMP data from the same season.

In fact, it appears from this intercalibration that differences generated during laboratory and instrumental methods are probably greater than the differences between the sampling systems.

The ratios of the dissolved to particulate concentrations between the systems was different for some of the 1996 compounds, but in the 1997 intercalibration the ratios are very similar. This probably reflects improved laboratory methods used in the extractions of the XAD absorbent. This indicates that the differences between the glass fiber particulate filters are not as great as originally believed, and in fact the filters seem to have very similar properties.

INTRODUCTION

The sampling and analysis of large volumes of water for trace organic compounds (chlorinated pesticides, OCs, polychlorinated biphenyls, PCBs, and polynuclear aromatic hydrocarbons, PAHs) is a difficult task; there are only a few research groups in the world which routinely undertake this (e.g., deLappe et al. 1983, Sarkar and Sen Gupta 1989, Hinckley and Bidleman 1991, Cruz et al. 1993, Iwata et al. 1993, Kelly et al. 1993, Schreitmuller and Ballschmiter 1995, Petrick et al. 1996). There are currently three absorbents used for the analysis of the dissolved fraction of large volumes (100 liters or greater) of water for trace analysis: 1) liquid-liquid extraction, where water is run through an organic solvent and the non-polar compounds partition from the water into the organic phase. 2) XAD resin, where the water samples are run through a column filled with an organic resin (or XAD-2) that absorbs the non-polar compounds, which are then eluted off the column using an organic solvent. 3) Polyurethane foam (PUF), where large volumes of water are pumped through PUF plugs and then the PUF is extracted with organic solvents. One aspect all of these sampling schemes share is the pre filtering of the

particulate fraction of the water prior to the absorbent. In general this is done using a glass fibre filter of 0.3-1 μM (either a flat or cartridge filter).

One of the most important quality control parameters involving the analysis of water is a careful characterization of the absorbent. In general this is accomplished three ways: 1) Direct laboratory experiments, where water is spiked with a known concentration of the compounds of interest and then the amount absorbed from the water is calculated (percent recovery). 2) The comparison of one absorbent with another absorbent that has been well characterized (e.g. PUF vs. liquid-liquid). 3) Determination of replicate analysis of one water sample, this provides information on the variability of the sampler.

Both liquid-liquid and the XAD resins have been extensively validated for the analysis of sea water for organic contaminants in a variety of field and laboratory studies (Ahnoff and Josefsson 1974, Osterroht 1974, Otson and Williams 1981, Sarkar and Sen Gupta 1989, Cruz et al. 1993, Kelly et al. 1993, Petrick et al. 1996).

There are less validation studies for PUF water samplers. Musty and Nickless (1974) spiked tap water with chlorinated pesticides and PCBs at $\mu\text{g/l}$ concentrations and determined their recoveries in a six different PUFs. They characterized the foam by its ability to absorb methylene blue. They found recoveries $> 90\%$ in foam which strongly absorbed methylene blue (Musty and Nickless 1974). deLappe et al (1983) in a field experiment compared the recoveries of PCB, OCs, and PAHs in PUF to the recoveries found in liquid-liquid extractors. In general the concentrations in PUF plugs agreed well with those from the liquid-liquid extractors. In addition, deLappe et al. (1983) analyzed individual foam plugs in series (five plugs were used in series) and found the breakthrough of the majority of analytes was $<10\%$. Validation of PUF as an air sampler has been more rigorous; (Nerin et al. 1995) found excellent recoveries of OCs in foam and found better precision in the PUF than in XAD resins for air sampling.

During the period 1993 through 1996 the sampler used in the Regional Monitoring Program (RMP) was a modification of deLappe et al. (1983). The sampler consisted of four PUF plugs in series; each plug is held in a separate cartridge that directs the water stream exclusively through the plugs, eliminating waterflow around the plugs. This sampler had not been used in intercalibration exercises, nor was any laboratory studies been performed. However, the design of this sampler is excellent for the determination of the capacity (or breakthrough) of compounds because each PUF plug can be analyzed separately and the breakthrough determined.

In 1996, a decision was made to switch from the Bodega Bay Institute's system BBI PUF sampler (BBI) to a commercially made XAD sampler (AXYS environmental, Sydney British Columbia) for the 1997 RMP. Prior to switching systems an intercalibration program was designed to examine the similarities/differences in the data generated by the two systems.

The BBI system consists of a Teflon impeller pump with 3/4 inch Teflon tubing, a flat glass fiber filter (GFF)(293 mm x 1 µm), and four polyurethane foam plugs mounted in series (to prevent channeling) which adsorb the dissolved material. No flow controller is used in this system (i.e., at low particulate loads on the GFF the flow is rapid, as the filter clogs the flow decreases). It is well known that flow has an important effect on the absorption of contaminants to the absorbent (Jarman et al. 1998).

The custom manufactured AXYS system consists of a constant flow PEEK gear driven positive displacement pump, 1/2 inch Teflon tubing, 1 µm glass fiber cartridge (GFC) particulate filter, and two parallel Teflon columns filed with -2 resin (parallel columns were employed to increase total flows). The use of the GFC system was chosen because of its high capacity for collecting particulates in water with high total suspended solids.

METHODOLOGY

Sample Locations

The intercalibration was designed to sample four different possible water parameters that might be encountered during an RMP sampling event: 1) low contaminant concentration sites; 2) high contaminant concentration; 3) low and 4) high salinity locations.

Samples were collected at six locations, 3 in 1996 and 3 in 1997. In 1996 Redwood Creek (BA40) and Coyote Creek (BA10) were sampled on June 27th, and Golden Gate (BC20) was sampled on June 28th. In 1997 the Sacramento River (BG-20) and San Joaquin River (BG-30) were samples on January 29th and Standish Dam BW-10 was sampled on January 22nd. Redwood Creek, Coyote Creek, and the Standish Dam site have been shown to have high concentrations of contaminants in previous RMPs; Golden Gate has low concentrations of contaminants and high salinity, and the river sites have low salinity

Samples were collected simultaneously with the intakes of the two sampling tubes less than one meter apart. All samples were collected as in previous RMPs (except the Golden Gate site of 1996 where approximately 200 L of water was collected, rather than the usual 100L.

Axys Sampler operation: See SOP Environmental and Geochemical Analytical Laboratory, University of Utah; EGAL SOP revised 1/15/97 (attached as appendix).

Analysis of Samples at The University of Utah

The extracts in this intercalibration were analyzed separately by the BBI and the University of Utah (U of U) i.e., the XAD samples were extracted and quantified at the U of U, and the BBI samples were extracted and quantified by the BBI. This is **not** normal protocol for RMP, and may explain some of the differences seen (see below).

Laboratory extraction: For complete methods see SOP Extraction of Water with -2 Resin for the San Francisco Bay Project, Environmental and Geochemical Analytical Laboratory, University of Utah, EGAL SOP revised 11/12/97 (attached as appendix).

Briefly, the method is:

Filter cartridges: Samples were spiked with surrogate recovery standards, and the cartridges were extracted in gravity flow columns sequentially with methanol and methylene chloride. The extractions were combined and the phases were separated. In addition, to check the method recovery, a post extraction rinse (PER) was taken of each cartridge (pesticides only). This was essentially a second complete extraction of the cartridge.

XAD columns: Each of the two columns (samples consist of two parallel XAD columns) was spiked with extraction surrogates, and eluted in reverse with methanol and methylene chloride in a method similar to the filter cartridges. The separate extracts were then combined and separated into two fractions on Florisil.

Both these extraction methods were based upon standard EPA and AXYS extraction protocols.

The extracts were subjected to Florisil column chromatography resulting in two fractions, a PCB/aliphatic and Pesticide/aromatic fractions. In the 1996 intercalibration, a polar third fraction, which contains Diazinon and Dacthal, was not taken, however, it was added for the 1997 samples.

Blanks

Blanks of the XAD columns and GFCs were transported with the sample columns and filters on the both the 1996 and 1997 cruises. The blank data is reported in Tables 13-18.

RESULTS

Pesticides

Levels

Result of the pesticide concentrations for the 1996 and 1997 intercalibration and various RMP cruises are presented in Tables 1 and 2. The past RMP cruises were chosen to compare temporal and seasonal values. (The results of the Coyote creek dissolved intercalibration cruise, is the level in only one of the two XAD columns because one column extract was lost.)

The pesticide levels (total of particulate and dissolved) of the 1996 intercalibration cruise are near, or within the range of the previous values generated by the BBI system during the intercalibration and previous RMPs for all three stations (Table 1). For example, total DDE levels for Redwood creek range from 82 to 140 pg/L (cruises 5,8-9), and are 69 pg/L during the intercalibration for the XAD sampler and 78 pg/L for the BBI system.

DDE levels at the Golden Gate range from 11 to 61 pg/L during the RMP, and are 36 pg/L (XAD) versus 85 pg/L (BBI) for the intercalibration. The BBI value of 85 pg/L is one of the highest values for DDE ever reported at Golden Gate.

In general, however, the agreement between systems, even at very low levels, is very good. For example, Chlordane levels for both systems were very similar at all stations (Figures 1abc).

The Figures are presented on a log scale to allow comparisons between the DDT compounds (p,p'-DDT, p,p'-DDE, and p,p'-DDD), HCH compounds (α -HCH, β -HCH, γ -HCH, and δ -HCH), Chlordane compounds (*cis*-Chlordane, *trans*-Chlordane, *cis*-nonachlor, *trans*-nonachlor, oxychlordane, heptachlor epoxide, and Heptachlor), and Diazinon (1997 only).

The 1997 pesticide intercalibration data, along with RMP data for cruises 7 (winter), 10 (winter), and 11 (spring) are presented in Table 2; the total (dissolved plus particulate) concentrations of the pesticides are presented in Figures 2abc.

As with the 1996 pesticide intercalibration there is no clear differences between the data generated in previous RMPs and either the XAD or BBI sampler in the 1997 intercalibration. For example, levels of Diazinon in the Sacramento River are similar between the XAD and BBI sampler during the intercalibration, higher in the San Joaquin BBI samples, and higher in the XAD sample from the Standish Dam (Figures 1abc). However, the intercalibration samples for both the XAD and BBI sampler are similar to past RMP cruises. An exception to this is the 1997 San Joaquin samples which are higher (in both samplers) than past winter or spring RMP cruises (as is the values for sum DDTs) (Figure 2b).

Levels of other pesticides (HCHs and Chlordanes) are similar for both the intercalibration and past RMPs (Figures 2abc).

Ratios

One of the major differences in the systems is the particulate filter. As mentioned above, the AXYS system had a wound glass cartridge system (GFC) and the BBI system uses a flat glass fiber filter system (GFF).

In an attempt to examine the partitioning between the dissolved and particulate phases, the ratio of the pesticides (dissolved/particulate concentration) for the 1996 and 1997 intercalibration were compared (Tables 3 and 4).

In 1996 some of the ratios of dissolved to particulate concentrations were lower in the XAD sampler; however, many of the ratios were similar (e.g., DDTs, Chlordanes-except HE) (Table 3). In particular, the ratios of the HCH compounds are much lower in the system. However, the ratios of the XAD sampler are within those values generated in cruises 5, 8, and 9.

In the 1997 intercalibration the ratios are more similar between the systems, and are often higher in the XAD system (Table 4). In particular, with the HCH compounds there is a

reverse of the 1996 data in that the XAD ratios are higher than the BBI ratios. In addition, the BBI DDE ratio for Standish Dam is 1000, which is probably a data error.

At this time it is impossible to determine why the ratios were low in the 1996 cruise. It is very probable though, that the laboratory method used for extraction of the pesticides on the resin has improved through method development, resulting in higher ratios in 1997. We will continue to examine the dissolved/particulate ratio in the future to attempt to clarify this trend.

Blanks Blanks for both the 1996 and 1997 intercalibration show no or very low pesticide contamination (Tables 13 and 14).

Polychlorinated Biphenyls

In 1996 the levels of polychlorinated biphenyls in the blanks were very high in both the filters and columns (Table 15). This contamination was traced to the ventilation air in the Applied Science building at UCSC (we have now moved into a cleaner laboratory at the U of U and do not have problems with blank contamination). Unfortunately, this source was not identified before the intercalibration samples were extracted. This necessitated subtracting the PCB values for the blanks from the concentrations in the intercalibration samples, a practice not usually employed in the RMP trace organic water samples.

As with pesticides, except for the values of PCBs in the Golden Gate site, most of the concentrations in the AXYS sampler fell between previous RMP values (Table 5). For example, Σ PCBs ranged from 980 to 2700 pg/L during cruises 5, 8, and 9 at Redwood creek, and values of Σ PCBs during the intercalibration were 1100 pg/L (Table 5). Levels of Σ PCBs at Coyote creek ranged from 1200 to 6800 pg/L during the RMP and were 1500 pg/L during the intercalibration.

High levels in the blanks make it impossible to compare values in the Golden Gate site. Also, because of the uncertainty associated with the blank corrected values, PCB congener profiles and dissolved/particulate ratios were not compared in the 1996 site.

The 1997 PCB intercalibration did not have blank problems, and data is presented in Table 6. The sum PCBs (sum of the congeners for both the dissolved and particulate fraction) is presented in Figures 3abc. The sum PCB values for the XAD sampler are similar to previous cruises for the same season for all three sites. However, sum PCB data from the BBI sampler is much higher than in previous RMPs and the 1997 intercalibration sites (Figures 3abc). In addition, the congener profiles from the BBI 1997 samples are different than the sampler or previous RMP data; the 1997 BBI data has a greater percentage of higher chlorinated biphenyls (or lower percentage of lighter chlorinated biphenyls)(Figures 4abc).

This bias probably results from differences in analytical methodology, rather than differences in the sampler characteristics. This conclusion is supported by two observations. First, as mentioned in the methods, the PCB data in the intercalibration was generated entirely by the Bodega Bay Institute, and this is not normal protocol for the RMP; in all of the past RMPs all the water sample chemical extracts were analyzed by the University of Utah (or prior to 1997 by the same group at the University of California Santa Cruz). Secondly, it has been noted by Davis et al. (1997) there is a great deal of analytical variation between laboratories in the analysis of PCBs in water extracts from RMP sites.

Ratios

The clean blank in the 1997 intercalibration allow the comparison of dissolved to particulate ratios between the samplers (Tables 7 and 16). There is no clear trend between the XAD and BBI samplers as to higher or lower ratios. In other words, sometimes the particulate/dissolved ratio is sometimes higher in the XAD sampler, and sometimes lower. There is no discernable pattern with regard to degree of chlorination, nor is there a pattern relating to high or low concentration sites (i.e., the river sites versus the Standish Dam)

As far as PCBs are concerned, the dissolved/particulate ratio is very similar between samplers.

Polycyclic Aromatic Hydrocarbons (PAHs)

The levels of PAHs collected during the intercalibration and past RMP cruises are reported in Tables 8 (1996) and 9 (1997). Levels of PAHs in the blanks (Tables 17 and 18) were very low in both the columns and glass fiber filters, except Naphthalene and 2-Methylnaphthalene in the blank in 1996.

The concentrations of the PAH compounds between the samplers used during the intercalibration and for the previous RMP are similar for most of the compounds analyzed (Tables 8 and 9; Figures 4abc and 5abc).

In the 1996 intercalibration the levels of PAHs in the XAD sampler were slightly lower, in general, than in those reported by the BBI sampler in the intercalibration or during RMP cruises 9 and 8 (levels of PAHs compounds during cruise 8 in the south bay were some of the highest recorded to date in the RMP).

The levels at the Golden Gate site, which are some of the lowest in the RMP, are in general very comparable, with some exceptions (e.g., fluoranthene). However as mentioned above, PAH levels are generally lower in the AXYS system.

However, in the 1997 intercalibration the value of individual PAHs are more similar between the systems.

The sum PAHs for the 1997 intercalibration, and RMP cruises 7, 10, and 11 are summarized in Figures 5abc. Both the Sacramento River and Standish Dam sum PAHs are similar between the samplers and within values generated in previous RMPs. Values for the sum PAHs for the San Joaquin River samples are elevated in BBI sampler (Figure 5b). This is probably due to the extremely high value reported for benzo[g,h,i]perylene (13,000 pg/L).

Profiles of the major individual PAHs for cruises 10, 11, and the intercalibration show similar profiles for most compounds except for 1-methylnaphthal, 2-methylnaphthal, fluorine, and as mentioned above, benzo[g,h,i]perylene (Figure 6abc). Similar to the PCBs, these discrepancies are probably a result of difference in the method of quantification, rather than inherent differences between samplers.

Ratios

Ratios of the dissolved to particulate concentration for RMP cruises 8 and 9 and the 1996 intercalibration are presented in Table 10, and cruises 7, 10, 11, and the intercalibration for 1997 in Table 11. The ratios are very similar between samplers; for example ratios high in the RMP are often have high ratios in the intercalibration (e.g., phenanthrene (s)); and similarly low ratios in the RMP are often accompanied by low ratios in the intercalibration (e.g., Benzo[e]pyrene). The extremely high ratios of Naphthalene are probably indicative of contamination (possibly in the resin).

Table 12 shows the dissolved/particulate ratio for two sites (Golden Gate and Point Pinole) for cruises 6 through 13 show the similarities between the systems during actual RMP cruises. The BBI system was used for cruises 6 through 12, and starting with cruise 13 the XAD system was used. The ratios for a particular season (e.g., winter) are very similar.

Quality Control

Besides blanks, two other QA protocols were investigated during the intercalibration. The first was the checking of efficiency of the XAD columns to extract the analyte from water; this was done by attaching a second column in series with the first during sampling at Redwood creek.

A second QA check was performed by extracting the particulate filter two separate times with solvents to check the extraction efficiency of the solvents. This was done because surrogates spiked onto the filters do not truly mimic compounds that may be attached or “trapped” to particulates or solids.

Serial Columns

The concentrations and breakthrough for the pesticides and PAHs in serial columns from Redwood creek are presented in Tables 19 and 20. Most of the breakthroughs for the pesticides are less than 20%. Only DDE has significant breakthrough (34 and 44 %) in both (columns 1 & 2) “after” columns.

Excluding the naphthalene compounds (because of their high blanks values) the PAH compounds have a breakthrough percentage of between 2 and 36%. Most of the compounds have a breakthrough less than 20% (Table 20).

Since this type of experiment has never been done before in the RMP, interpretation of the data is difficult. As a general rule, recoveries of surrogates in extractions should be between approximately 50-120%, but this is widely considered very liberal.

To better interpret this data we recommend that serial recovery columns be analyzed at least once a year in the RMP. In addition, recovery data from the BBI sampler consisting of the analysis of individual plugs will allow comparisons to this data.

Post extraction rinse

Table 21 presents the data for pesticides from the post extraction rinse (PER) of the glass fiber filter for the Coyote creek and Redwood creek intercalibration. Except for DDE and some of the Chlordane compounds, greater than 90% of the pesticides are extracted in the first. Because of the carry over of DDE in the PER, additional extractions will be performed on filters in the future. This method will also then be validated for PCBs, pesticides, and PAHs

CONCLUSIONS

In general, levels of pesticides, PCBs, and PAHs are similar between the XAD sampler and BBI sampler. In addition, comparison of temporal trends in past RMPs show the data generated by the sampler to be very similar to past RMP data from the same season.

In fact, it appears from this data that differences generated during laboratory and instrumental methods are probably greater than the differences between the sampling systems.

The ratios of the dissolved to particulate concentrations between the systems was different for some of the 1996 compounds, but in the 1997 intercalibration the ratios are very similar. This probably reflects improved laboratory methods used in the extractions of the absorbent. This indicates that the differences between the GGF and the GFC particulate filters are not as great as originally believed, and in fact the filters seem to have very similar properties.

High blank levels of PCBs from the UCSC laboratory prohibit detailed (e.g., congener profiles) comparisons in 1996, but by 1997 this contamination has been addressed and blanks were found to be clean. High naphthalene concentrations were found in some blanks, but not all, and this contamination will be further investigated. Other than the high PCB (in 1996 only) and naphthalene blank levels, all other compounds were very low in both the glass fiber filter and columns.

Data from the serial columns indicate some breakthrough (generally < 20%) from the first column into the second. We recommend continuing this practice to better quantify breakthrough.

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