**QUALITY ASSURANCE PROJECT PLAN**

**United States Environmental Protection Agency GRANT #GL00E76601-4**

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**INTEGRATED ATMOSPHERIC DEPOSITION NETWORK**

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# 1. PROJECT DESCRIPTION

Introduction

Evidence has mounted that atmospheric transport and deposition is an important pathway by which certain toxic organic chemicals reach the Great Lakes **(**Venier et al., 2008a, Venier et al, 2008b, Sun et al., 2006a, Sun et al., 2006b, Buehler et al., 2001, Cortes et al., 2000; Swackhamer, D. L., 1988, Eisenreich et al., 1981; Hillery et al., 1997; Murphy et al., 1981; Rice et al., 1986, and Simcik et al., 1999). To evaluate the sources and fate of toxic organics in the Great Lakes, a mass balance approach has been selected (Strachan and Eisenreich, 1988; U.S.EPA/GLNPO, 1985). This approach requires the determination of system inputs, internal transformations and storage, and outputs. If system inputs, less transformation and storage, equal outputs then the system can be assumed to be understood and future contaminant concentrations can be accurately modeled. Remediation strategies can then be devised to prevent toxic contaminants from reaching dangerous concentrations in water and biota.

In an effort to determine the status, change, and trends of toxic organics in the Great Lakes, the United States and Canada are cooperating on a joint monitoring program titled the Integrated Atmospheric Deposition Network (IADN). The intent of the network is to collect and evaluate pollution data in the atmosphere (airborne vapor, airborne particles, and precipitation) at a regional level of detail. Present organic toxics of interest are chlorinated pesticides, including alpha- and gamma-hexachlorocyclohexanes (HCH), polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and trace metals including lead. Metal analyses were stopped by the U.S. EPA in January 2001. Analysis of polybrominated diphenyl ether (PBDE) and other flame retardants was started in January 2003 with method development; actual flame retardant analysis and data reporting started in 2005.

IADN was created in response to the Great Lakes Water Quality Agreement (Annex 15). This annex includes a research phase and a surveillance and monitoring phase. IADN is implemented through a binational committee which sets program objectives and implementation schedules. The data collection effort of the monitoring network is focused on five master stations, one on each lake. Master stations include more intensive monitoring activity and have space available for other research activities. Presently, there are three master stations in the U.S. and one in Canada are

collecting data. The three U.S. master stations are at Eagle Harbor, Michigan, Sleeping Bear Dunes, Michigan, and Sturgeon Point, New York. The Canadian master station is at Point Petre. The fifth master station at Burnt Island (near Lake Huron) was closed in 2002. Table 1.1 and Table 1.2 describe the different activities from 1993 to 2017 and current and future plans. Table 1.3 describes the equipment, frequency and the types of samples collected at each of the United States master stations. Similar samples are being taken at the Canadian site. As the program continues, satellite stations were added to each lake in order to provide additional sensitivity and statistical reliability in the data. Routine monitoring (less intensive) occur at the satellite sites. In the U.S., these satellite stations are in Chicago, Illinois, and Cleveland, Ohio. The locations and the IADN criteria for site selection are given in Section 17.1. All U.S. master and satellite sites are currently fully operational.

The data collection methods implemented for IADN are the most currently accepted methods for the pollutants of interest at low-level concentrations. QA/QC samples and various QA techniques are used to control and assess various phases of the measurement uncertainty. These uncertainty estimates can then be used to evaluate the various data collection methods. This QAPjP includes measurement quality objectives for the phases of work.

Project Schedule

IADN sampling and analysis occur year round since initial funding for this project was received in 1991, at Illinois State Water Survey. The first QA report was submitted in 1992. The project came to Indiana University in August 1994. The yearly updates of the QA report are available from 1996 and will continue until the conclusion of the project. Figure 1.1 represents a timetable for various Illinois State Water Survey (ISWS) and Indiana University (IU) activities.

| | 1991 | 1992 | 1993 | 1994 | 1995 | 1996 | 1997 | 1998 | 1999 | 2000 | 2001 | 2002 | 2003 | 2004 | 2005 | 2006 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Installation and Operation:** | | | | | | | | | | | | | | | | |
| **Master Stations:** | | | | | | | | | | | | | | | | |
| Lake Superior: Eagle Harbor | | | | | | | | | | | | | | | | |
| Lake Erie: Sturgeon Point | | | | | | | | | | | | | | | | |
| Lake Michigan: Sleeping Bear | | | | | | | | | | | | | | | | |
| **Satellite Stations** | | | | | | | | | | | | | | | | |
| Lake Michigan: Chicago | | | | | | | | | | | | | | | | |
| Lake Superior: Brule River | | | | | | | | | | | | | | | | |
| Lake Erie: Cleveland | | | | | | | | | | | | | | | | |
| Lake Ontario: Point Petre (QC sampling at Canadian Master site) | | | | | | | | | | | | | | | | |
| **Method Developments** | | | | | | | | | | | | | | | | |
| Sampling Methods | | | | | | | | | | | | | | | | |
| Analytical Methods | | | | | | | | | Precip. | | | | | | | |
| Routine Analysis | | | | | | | | | | | | | | | | |
| **Reporting:** | | | | | | | | | | | | | | | | |
| QA Reports | | | | | | | | | | | | | | | | |
| Data Reports and publications | | | | | | | | | | | | | | | | |
| | **ISWS** | **ISWS** | **ISWS** | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** |

**Figure 1.1.** Project Schedule

| | 2007 | 2008 | 2009 | 2010 | 2011-2014 | 2015-2019 |
|---|---|---|---|---|---|---|
| **Installation and Operation:** | | | | | | |
| **Master Stations:** | | | | | | |
| Lake Superior: Eagle Harbor | | | | | | |
| | | | | | | |
| Lake Erie: Sturgeon Point | | | | | | |
| | | | | | | |
| Lake Michigan: Sleeping Bear | | | | | | |
| | | | | | | |
| **Satellite Stations** | | | | | | |
| Lake Michigan: Chicago | | | | | | |
| | | | | | | |
| Lake Superior: Brule River | | | | | | |
| | | | | | | |
| Lake Erie: Cleveland | | | | | | |
| | | | | | | |
| Lake Ontario: Point Petre | | | | | | |
| (QC sampling at Canadian | | | | | | |
| Master site) | | | | | | |
| | | | | | | |
| **Method Developments** | | | | | | |
| Sampling Methods | | | | | | |
| | | | | | | |
| Analytical Methods | | | | | | |
| | | | | | | |
| Routine Analysis | | | | | | |
| | | | | | | |
| **Reporting:** | | | | | | |
| QA Reports | | | | | | |
| Data Reports and publications | | | | | | |
| | **IU** | **IU** | **IU** | **IU** | **IU** | **IU** |

**Figure 1.1.** Project Schedule (continued)

Scope of Work

*Introduction*

IU provides assistance to the IADN program through subtasks related to sample collection, sample analysis, quality assurance, methods development, data management, data interpretation, communication, and publication of results.

*Sample Collection*

Sampling will continue throughout the year at the master sites located at Eagle Harbor (Keweenaw Co.), Michigan, on Lake Superior; at Sturgeon Point, New York, on Lake Erie; and at Sleeping Bear Dunes National Lakeshore, Michigan, on Lake Michigan. Sampling began at two satellite sites located at Brule River, Wisconsin, on Lake Superior and the Illinois Institute of Technology, Chicago, Illinois, on Lake Michigan in April 1994 and January 1993 respectively. In 2002-2003 the Brule River site was closed, and all of the sampling gear moved to another satellite site at Cleveland on Lake Erie.

Samples of precipitation and airborne particles and vapors are collected. Precipitation samples are collected in automated wet-only samplers manufactured by the MIC Co. of Thornhill, Ontario. Two of these samplers are in operation at the Lake Superior, Lake Michigan, and Lake Erie master sites, collecting routine samples, duplicate samples or field blanks once a month. Particles and vapors are collected using modified Anderson high-volume air samplers to draw air through a quartz fiber filter and XAD-2 resin. Duplicate samplers operate at each master site, sampling for 24 hours once every 12 days (Basu et al, 2010). A standard high volume sampler collected a sample of ambient particles every sixth day for determination of the concentrations of total suspended particulate matter (TSP), and non-volatile elemental and organic carbon (TOC), but this part of the project was discontinued in August 1996. The proposal and approval of stopping the TSP/TOC sampling and analysis can be found in Appendix section 17.4.

*Sample Analysis*

Methods are implemented at Indiana University (IU) to analyze all target organic compounds. Target organic compounds include PCBs, selected chlorinated pesticides, PAHs, PBDEs and other brominated flame retardants (Team IADN, 2013; Ma and Venier, 2013). When appropriate, some samples like particle samples on quartz fiber filters were composited before analysis. This was done especially for PCB analysis. IU stopped compositing the filters because it was not possible to correlate the composite data with daily meteorological information and with corresponding gas phase data. About 50% of the information was lost this way especially for PAHs. Analyzing each filter extract, without compositing, increased the workload and the cost of analysis. To compensate this extra analytical cost, IU proposed to drop the PCB analysis on filters because 90-95% of the atmospheric PCBs are in the gas phase. Angela Bandemehr approved this on December 3, 1996. At the same time, the HCB and p,p'-DDE analyses on particle samples were also discontinued. IU continued analysis of individual filters for pesticides and PAHs only. The proposal, logistics and approval of these changes can be found in section Appendices 17.5 and 17.6.

Pesticide analyses on filters from Eagle Harbor, Sleeping Bear Dunes, and Sturgeon Point were stopped in January 2004. However, IU continued the analysis of pesticides on filter samples from Chicago and Cleveland. The proposal, logistics and approval of these changes can be found in section Appendices 17.10.

*Method Development*

The Implementation Plan for IADN calls for continued development and improvement of sampling and analytical methods. IU will direct its effort primarily toward compounds targeted for analysis.

*Data Management and Interpretation*

IU documents sampling information and analytical results in a computer database for subsequent analysis. Increasing emphasis is placed on summarizing and interpreting the data in terms of atmospheric fluxes to the lakes and probable contaminant sources as additional data are accumulated.

*Reporting*

IU provides summaries for each sampling site detailing equipment in operation, air and precipitation samples collected, method changes, and problems encountered. In addition, we publish and provide periodic updates and interpretation of our chemical data in scientific journals.

**Table 1.1.** Activities from 1993-2003 are described below. The project was established in 1994. Now it is in maintenance stage.

| 1993-1994 | 1996-1997 | 1997- 2001 | 2002-2003 | 2003-2017 |
|---|---|---|---|---|
| Began sampling operations at two new US satellite stations Chicago and Brule River | Updated estimates of atmospheric deposition | Updated estimates of atmospheric deposition | Brule River was closed and a new urban site at Cleveland started | |
| | Reviewed IADN research activities | Reviewed IADN research activities | Continued sampling and analysis | Continued sampling and analysis |
| | Reviewed/modified target compound list | Reviewed/modified target compound list | Usual activities of reporting , publication and presentation | Usual activities of reporting, publication and presentation |
| | Data report | Data report and publications | Reviewed/modified target compound list; added PBDE (method development) | Reviewed/modified target compound list; added new flame retardants |
| | Annual QC report | Interlaboratory comparison studies | Interlaboratory comparison studies | Interlaboratory comparison studies |
| | | Point Petre collocation site continued | Point Petre collocation site continued | Point Petre collocation site continued |
| | Point Petre collocation site started | Sharing information with other organizations | Sharing information with other organizations | Sharing information with other organizations; published a visualization website featuring IADN data |
| | | Generated loading report | Generated loading report | Generated loading report |

**Table 1.2.** Current and Future Plan of activities

| 2004-2011 | 2012-2014 | 2015-2019 |
|---|---|---|
| Continued sampling and analysis | Continued sampling and analysis | Continuous |
| Updated estimates of atmospheric deposition to the lakes and spatiotemporal trends in atmospheric concentrations | | |
| Reviewing IADN research activities | Continuous | Continuous |
| Data reporting<br>a) Field and Meteorological data<br>b) Organic data<br>c) QC report | <br>a) Field and Meteorological data<br>b) Organic data<br>c) QC report | <br>a) Within May following year<br>b) Within October following year<br>c) Within November following year |
| Interlaboratory comparison studies:<br>a) Point Petre data<br>b) Round Robin studies | Interlaboratory comparison studies:<br>a) Point Petre data<br>b) Round Robin studies | Within specifies time |
| Sharing information with other organizations | Continuous | Continuous |

**Table 1.3.** IADN Master Sites Equipment and Activity Description

| Description of Sampler | Number of Samplers | Use/Pollutants | Frequency of Sampling | Analysis | Lab[a] | Status |
|---|---|---|---|---|---|---|
| Precipitation | | | | | | |
| Aerochem | 1 | rain, pH, conductivity | 1/7 days | Nutrients, pH, inorganics, conductivity | 1 | Discontinued in March 1995 |
| Belfort/Nipher | 1 | rain | continuous | | 2 | Stopped in August 2004 |
| MIC | 2 | semi-volatile organics | monthly collection started in August 2004 | PCBs, Pesticides, PAHs, PBDE | 2 | Continuing monthly collection. 28 days collection was stopped in August 2004 |
| Air | | | | | | |
| TSP | 1 | mass | 1/6 days | TOC, TSP | 2 | Collection discontinued 1996 |
| Mod Hi-Vol | 2 | semi-volatile organics | 1/12 days | PCBs, pesticides, PAHs | 2 | Continuing |
| Dichotomous sampler | 1 | mass, coarse/fine | 96 hours per month* | Metals, PM-10 | 3 | Discontinued in 2001 |
| Meteorological | | | | | | |
| Campbell Tower | 1 | Temperature, relative humidity, solar radiance, wind speed, wind direction, and barometric pressure | continuous | | 2 | Continuing |

* US EPA stopped analyzing metals in November 1997. IU continued sampling and storing the samples for future use. IU extended the sampling time from 96 to 144 hours per month for remote sites (Eagle Harbor, Brule River, and Sleeping Bear Dunes) to ensure collection of sufficient samples so that the incoming laboratories do not have any detection problem. At the same time, Chicago sampling time was reduced from 96 to 72 hours because of high TSP values at Chicago. Sampling stopped in 2005.

[a] 1 = U.S. EPA Region V Central Lab; 2 = IU; 3 = U.S. EPA AREAL Lab

Note: It has been observed that new Hi vol samplers, when first used, emit PCBs and other contaminants from its surface, especially in summer. Data from these new samplers are usually higher than the data from the regular samplers. Sometimes, it takes up to one year to produce reliable data. Indiana University installed a new Hi volume sampler at Point Petre in November 2004, where one Hi-vol sampler was already collecting air samples for more than 4 years. At the beginning, the total PCBs collected by the new sampler was almost 3 times higher than the PCBs collected by the regular sampler. After 10 months, the PCB data by 2 samplers were comparable.

Storage places for the samplers and their different parts like filter holders, which are not in use, are also very important. They should always be stored in clean buildings, preferably newer buildings, where there are no big sources of contaminants. In the past, one Hi-vol sampler was stored in Q6 building at the Illinois State Water Survey where old PCB using transformers were stored. This sampler was installed in Point Petre in 1998. Unfortunately, abnormally high numbers of PCBs were noted. The data had to be discarded. (Basu et al, 2000)

## 2. PROJECT ORGANIZATION AND RESPONSIBILITIES

The Principal Investigator, Ronald A. Hites, is responsible for overall project administration. The Research Scientist, Marta Venier, is the co-PI, and she assists the PI in project administration and in research. The IU Project Director, Amina Salamova, is responsible for overall project management, including data generation, data review, and reporting. She also assists in project administration and in research development. Laboratory Manager, Karen Arnold, is responsible for reviewing sampling activities, scheduling analysis of samples for target compounds, and overall laboratory operations. Data Manager, Daniel Lehman, is responsible for reviewing obtained data, preparing data and QA reports, and maintaining the IADN database. Field Technician, Jim Bays, is responsible for managing the site operators, scheduling sample collection, receiving collected samples, and maintaining equipment at the sites. The site operator at each site oversees actual site operation and collection of samples. Laboratory Technician, Stephen Zulkowski, is responsible for laboratory sample treatment and analyses. Laboratory technician Kevin Romanak is responsible to data analysis, instruments maintenance and IADN related projects. Lauren Tucker is a part time laboratory assistant and helps with basic laboratory tasks. The organizational structure is shown in Figure 2.1.

The laboratories are located in the Multidisciplinary Science Building II, School of Public and Environmental Affairs, Indiana University, Bloomington, Indiana. The IU research team coordinates sample collection and method development, method improvement, and routine analysis of all target compounds.

Technical and QA lines of communications are shown in Figure 2.2. The EPA Project Officer has the overall responsibility for the QA of this project. Position titles and major responsibilities are listed below.

<u>GLNPO QA Project Officer</u>
The GLNPO Project Officer (PO) is the Agency official who initiates the grant, evaluates the proposal, and is the technical representative for EPA. The Project Officer is responsible for:

- Budgeting.
- Program planning, scheduling, and prioritization.
- Developing project objectives and data quality objectives.
- Ensuring that project meets GLNPO missions.
- Technical guidance.
- Program and data reviews including audits.
- Data quality.

- Final deliverables.

GLNPO QA Program Manager:
The GLNPO QA Program Manager (QAPM) is responsible for ensuring that each project funded by EPA satisfies the Agency's requirements for QA programs. The QAPM is responsible for:
- Offering guidance on QA techniques.
- Evaluating QA Project Plans (QAPjPs) and approving QAPjPs for the Agency.
- Assisting in the coordination of audits
- Conducts meeting
- Co-ordinate activities with the Canadian program Manager

Principal Investigator
The Principal Investigator (PI) will administer, manage, and monitor the grant. The PI is responsible for:
- Developing proposals, budgets, and the scope of the work.
- Developing the project objectives and data quality objectives.
- Overseeing implementation of data collection activities including site set-up, sampling, sampler calibrations, sample handling, scheduling of analysis, and data review.
- Data quality.
- Ensuring development of QA Project Plan.
- Preparing QA report and data reports.
- Following the scope of work and report deviations.
- Working closely with GLNPO project officer to develop overall goals of the project.
- Publications and presentation

Co-Principal Investigator
The co-Principal Investigator (co-PI) will aid the PI in administration, managing, and monitoring of the grant. The co-PI, jointly with the PI, is responsible for:
- Developing proposals, budgets, and the scope of the work.
- Developing the project objectives and data quality objectives.
- Overseeing implementation of data collection activities including site set-up, sampling, sampler calibrations, sample handling, scheduling of analysis, and data review.
- Data quality.
- Ensuring development of QA Project Plan.
- Preparing QA report and Data report.
- Following the scope of work and report deviations.
- Working closely with GLNPO project officer to develop overall goals of the project.
- Publications and presentation

Project Director
The Project Director is the technical expert of the project. Together with the PI and the co-PI, the person is responsible for:
- Developing proposals, budgets, and the scope of the work.
- Developing the project objectives and data quality objectives.
- Overseeing implementation of data collection activities including site set-up, sampling, sampler calibrations, sample handling, scheduling of analysis, and data review.
- Data quality.
- Ensuring development of QA Project Plan.
- Preparing QA reports and Data reports.
- Following the scope of work and report deviations.
- Working closely with GLNPO project officer to develop overall goals of the project.
- Overseeing overall project management and data generation process
- Reviewing data and reporting data to the EPA and Environment Canada; maintaining database
- Overseeing general laboratory operations and ensuring that established procedures (such as SOPs) are followed, and any deviations documented.

- Acquiring necessary personnel and equipment.
- Assisting PI with administrative tasks
- Assisting PI with research development
- Publications and presentations

Laboratory Manager
- Overseeing laboratory operations
- Overseeing implementation of data collection: sampling, sample handling, scheduling of analysis, and data archive.
- Developing the standard operating protocols for sampling and laboratory analysis.
- Maintaining up-to-date laboratory notebooks and sample logs and other appropriate record keeping.
- Entering data in the spreadsheets
- Preparing and updating SOPs

Data Manager
- Reviewing data
- Preparing data reports
- Archiving data
- Entering data in the spreadsheets
- Reviewing QA/QC data
- Quantitating data

QA/QC Monitor
The QA Coordinator is responsible for Data Management and QC coordination:
- Data quality evaluation
- Maintaining control charts
- Developing QAPjP
- Taking corrective actions based on audit.

Sampling Technician
The Sampling Technician will be responsible for:
- Maintenance and improvement of the sampling sites
- Working with site operators.
- Maintaining and calibrating samplers.
- Preparing sampling protocol and field report
- Preparing field SOPs
- Sending cartridges, filters, and other supplies to the sites and receiving samples
- Maintaining paper work and log regarding sampling
- Downloading Met Tower data from data logger

Laboratory Technicians:
- Extraction of samples and rotary evaporating
- Silica column fractionation
- Instrumental analysis of PCBs, pesticides, PAHs
- Maintenance of the GCs and GCMS
- Inventory of laboratory supplies
- Helping the Project Director with SOPs, and QC reports writing

Post-doctoral Associates and Doctoral Students:
The post-doctoral associates and the students are responsible for:
- Data interpretation and publications in scientific journals.
- Presentation in national and international scientific conferences.

Training:

The laboratory technicians were initially trained by the previous Deputy Principal Investigator, Dr. Ilora Basu. The training continues by the Project Director, Dr. Amina Salamova, and Laboratory Manager, Ms. Karen Arnold, whenever it is necessary. All laboratory personnel are internally trained by Indiana University personnel. The Field Technician, Jim Bays, was trained by Jim Osborne from the Illinois State Water Survey, where the IADN Project started. The site operators are trained by Jim Bays, Indiana University Field Technician. These trainings are a requirement for any new hire to work in IADN, they don't follow specific protocols and are tailored to the specific needs and tasks of the new personnel. For these reasons, a record of these trainings is not maintained. Occasionally, the technicians are trained by Agilent on instrumental analyses details. The records of instrumental analyses (certificates) are kept by program participants for their personal records.

**Figure 2.1.** Organizational Chart at IU

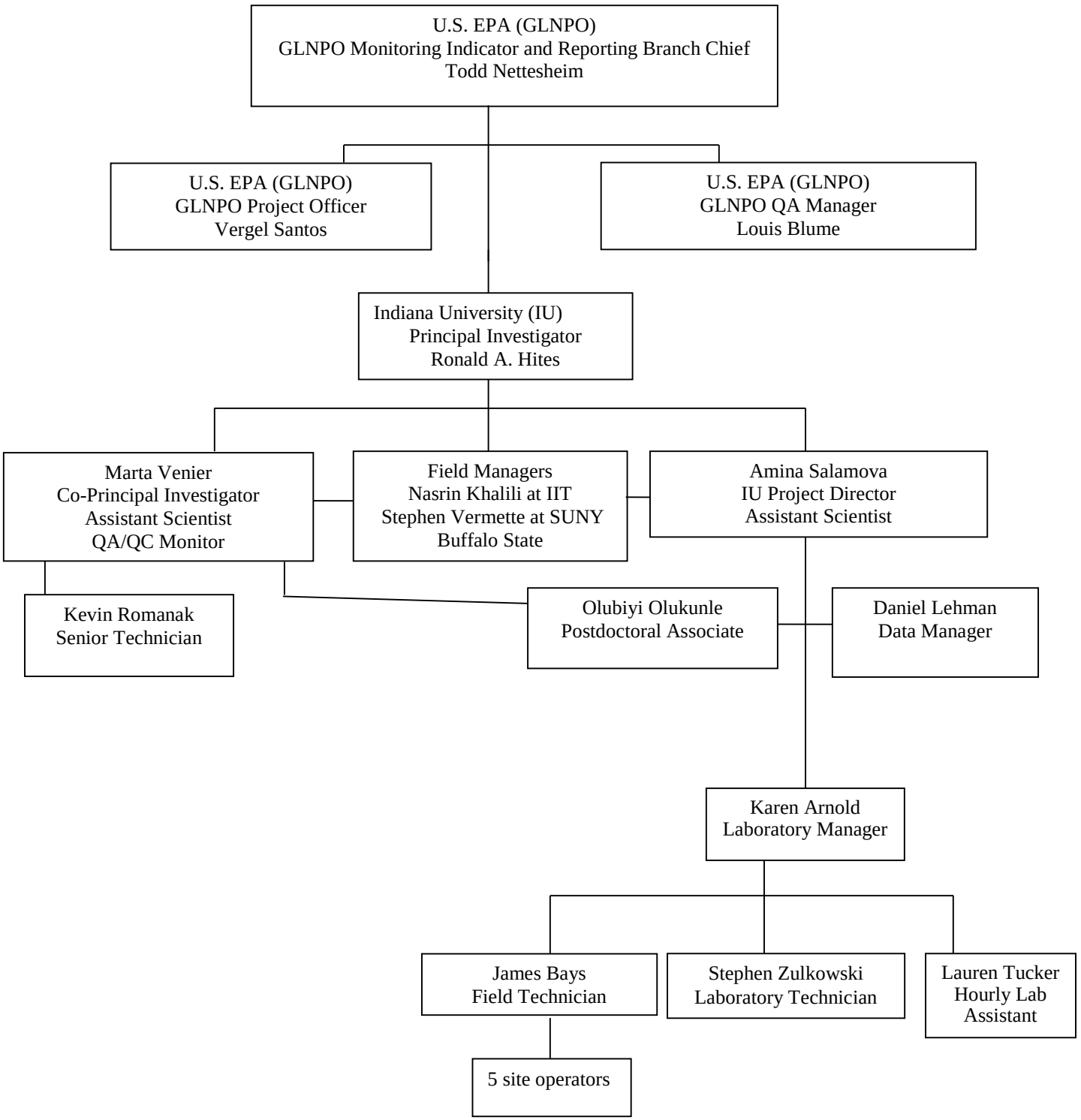
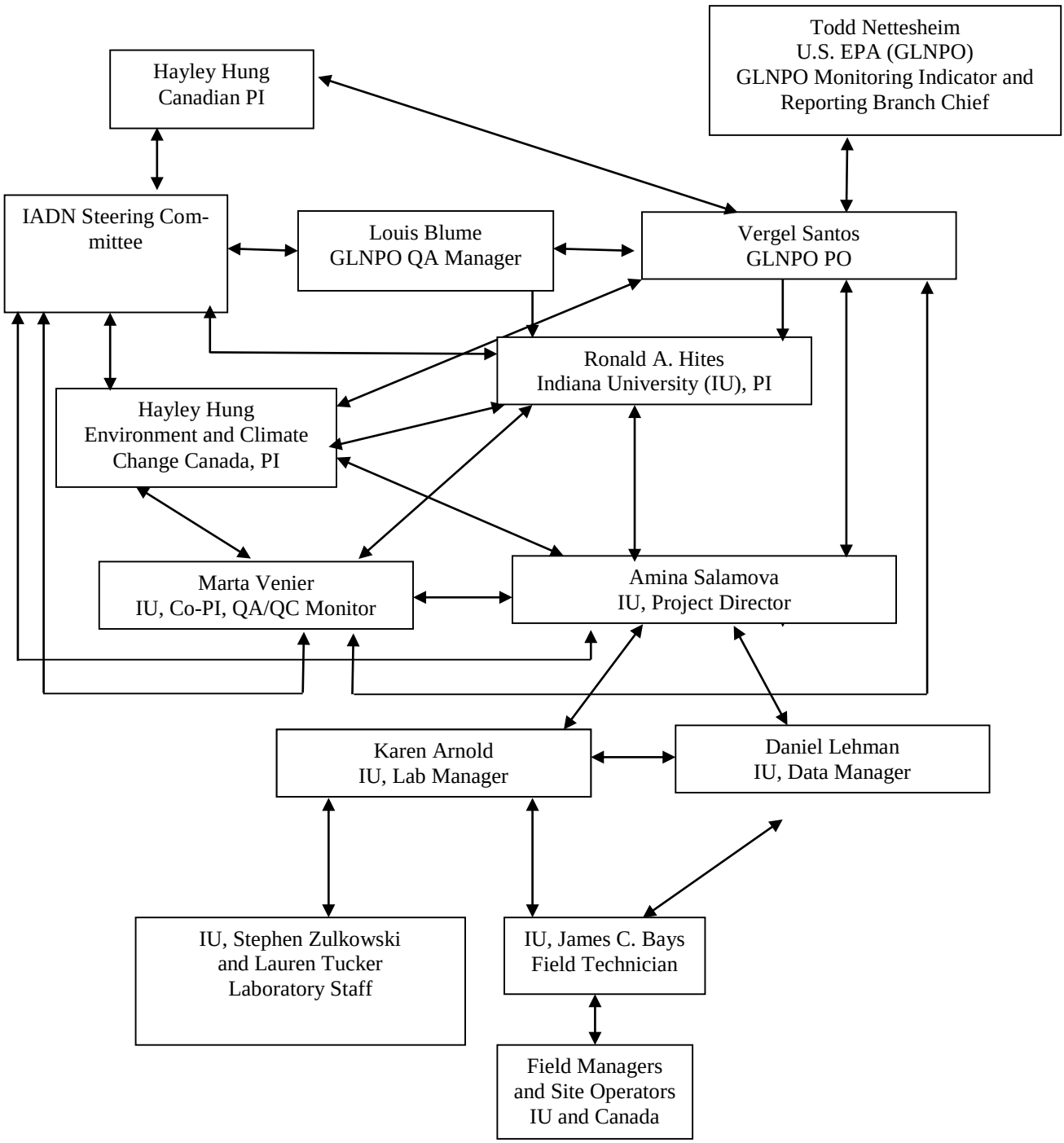


**Figure 2.2.** Technical and QA Lines of Communication for IADN Responsibilities



## 3. QUALITY ASSURANCE OBJECTIVES

This section describes the IADN QA program, which is designed to allow both control and assessment of measurement uncertainty during sampling, preparation, and analysis phases of the project.

Data Quality Objectives

This project is generally aimed at the Environmental Protection Agency's strategic goal of preventing water pollution and protecting aquatic systems so that the overall ecosystem health of the Great Lakes is improved. The strategic goal most related to IADN is to maintain or improve an average 5% annual decline for the long-term trend in average concentrations of toxic chemicals (PCBs) from the air from the Great Lakes Basin.

The expected outcomes from this project are, but are not limited to:

- Improved understanding of the atmospheric fate and cycling of priority toxic chemicals in the Great Lakes region;
- Improved understanding of the sources, trends, and loadings of chemicals of emerging concern entering the Great Lakes; and
- Better decisions by environmental managers in development and implementation of strategies to reduce the presence of priority toxic chemicals in Great Lakes air, water, sediment, fish, and other wildlife.

The IADN Data Quality Objectives (DQO) are to acquire sufficient quality assured data to estimate with a significant degree of confidence the loading to the Great Lakes basin of selected toxic substances (IADN Quality Assurance Plan 1994). The DQO is the overall uncertainty that the user is willing to accept in the result derived from the data while being able to make an informed decision. This means that both population and measurement uncertainties (field and analytical), to an extent, are understood. Adherence to accepted Standard Operating Procedures (SOPs) is an important part of the overall data quality of the IADN. The establishment of Measurement Quality Objectives (MQOs) is also used to control and assess the measurement uncertainties.

Measurement Quality Objectives

The majority of uncertainties occurring in the field can be alleviated by the development of detailed standard operating procedures (SOPs), an adequate training program at appropriate frequency, and a field audit program. SOPs have been developed and training has occurred. Field audits are being implemented on a regular basis, at least three times a year (spring, summer, fall) for each site. Results of all audits are documented. Measurement quality objectives are designed to control various phases of the measurement process and to ensure that total measurement uncertainty is within ranges prescribed by the DQOs. The MQOs can be defined in terms of data quality attributes: precision, accuracy, completeness, detectability, representativeness, and comparability. The first four can be defined in quantitative terms, while the latter two are qualitative. Tables 3.1 and 3.2 list MQOs for parameters included in this study.

*Precision*

Precision is a measure of the reproducibility among multiple measurements of the same property, usually under prescribed similar conditions. Quantitative measurements of precision include replicate field samples, replicate laboratory samples, and analysis by different methods for comparison. The applicability of these measurements is parameter dependent. In expressing overall variance of the measurement system, pooled data from collocated field duplicates (FD1/FD2) are used. Since field duplicates are routine samples in which the actual concentration is unknown, the estimate of overall variance may be influenced by concentration. The influence of concentration on variance is evaluated and the most appropriate approach to estimating overall variance is developed.

**Field Duplicate Samples (FD1/FD2):** Replicate samples are collected using collocated samplers concurrently. These samples contain a component of spatial uncertainty that cannot be separated from the measurement uncertainty with the present QA design. However, the precision estimate, when deducted from the laboratory duplicate precision estimate, can provide information on field sampling uncertainty. Acceptance criteria for organic compounds depend on the mass of compounds in the sample. For PCBs, total PCBs should be considered. The relative percent difference (RPD) should be <100%. For each pesticides and PAHs, RPDs should be < 100%. Field duplicates comprise, at a minimum, 5% of the field samples collected.

**Laboratory Duplicate Samples (LD1/LD2):** Samples are split to provide for duplicate laboratory analysis. Organic samples are split prior to extraction. Acceptance criteria for the organic compounds are listed in Table 3.1. Laboratory duplicates comprise at least 5% of collected field samples.

*Accuracy*

Accuracy is a measure of the degree to which a measurement or computed value reflects the true value of analyte present. Accuracy is assessed as the recovery of a standard reference material or surrogate/matrix spikes for organic analytes. Accuracy is also assessed using performance evaluation (PE) samples of "known" concentration that are either known or blind to the laboratory. Collocated field duplicate samples are analyzed at other laboratories or prepared samples are sometimes split and portions analyzed by other participating laboratories.

**Organic Analytes:** Accuracy for organic analytes is assessed by evaluation of recoveries using surrogate spikes (SS) in each sample and matrix spiked recovery samples (MS). Surrogate standards (laboratory surrogate spikes) are added to all samples prior to the extraction step to assess losses in sample preparation. A known amount of the surrogate standard is added directly to every sample and blank at the beginning of the extraction procedure. Thus, the recovery of every extraction can be estimated by the recovery of the surrogate spike. The matrix spikes (with PCB, pesticide, PAH, and PBDE standards) represent the actual analytical recovery for all target organic compounds. The acceptance criterion is that the recovery (R), relative to the certified or recovery standard must be between 50 and 130% for 70% of individual congeners included in each target group (PCBs, pesticides, PAHs, and PBDEs) and for the average.

Important sampling variables affecting accuracy are the flow rate of the high-volume samplers and the measurement of precipitation volume. Bias is the percentage difference between the nominal flow rate set when the instrument is initially calibrated and the actual flow rate determined when the calibration is checked.

**Blanks:** Field matrix blanks (FB) are used to assess the extent of background contamination present in the field. Laboratory matrix blanks (LB) are used to monitor the degree of background contamination introduced during the laboratory analysis and must meet the criterion of mass < LOD. Failure of this criterion should be addressed by running a second lab matrix blank and, if the criterion is still not met, find the source of contamination and eliminate it.

**Performance Evaluation (PE) Samples:** Accuracy is also evaluated by determining whether the concentrations of the PE samples are within the required acceptance windows. These windows have been established by the vendor supplying the PE material, in which case a certificate of analyses is included, or through the use of this sample by many laboratories using the same or a similar method, thereby establishing an accepted window. Values outside the acceptance windows could be justification for reanalysis. Accuracy is also measured by analyzing a custom made Common Reference Standard (CRS) for each group of analytes distributed to all participating laboratories by the QC officer (Canada).

*Completeness*

Completeness is the measure of the number of valid samples (meeting all QA requirements) obtained compared to the number required to achieve the objectives of the study. Overall completeness is the number of valid samples compared to the number of planned samples. Laboratory completeness is the number of valid samples obtained compared to the number of analyzed samples. Both types of completeness are reported for the project. Targeted threshold for completeness is 90% for field samples. As with the other data quality attributes, completeness can be controlled through adherence to the SOPs in order to minimize laboratory and sampling errors.

*Detectability*

Detectability refers to the determination of the low-range critical value of a characteristic that a method-specific procedure can reliably discern. The applicable detection limit for organics in IADN is the matrix specific method detection limit (MDL). MDL was calculated only for the first 2 years of the project, then it was switched to an Instrument Detection Limit (IDL). A diluted solution containing all compounds of interest is injected 7-10 times to determine the IDL. The standard deviations of measurements obtained in these repeated injections are calculated and multiplied by 3 to obtain the IDL for each compound. The IDL is assessed once every year.

*Limit of detection*

The limit of detection (LOD) is defined for the case where repeated analyses of field blanks show a positive response for the analyte. The LOD is then given by

$$LOD = C_b + t_{(n-1,\ 1-\alpha)} \times S$$

where $C_b$ is the average level for the field blanks, $S$ is the standard deviation of the replicate determinations, $t_{(n-1,\ 1-\alpha)}$ is the Student's $t$-distribution for $n$-1 degrees of freedom and 1-$\alpha$ is chosen to be 0.95 or 0.99. For the IADN, $t_{(n-1,\ 1-\alpha)}$ is assumed to be 3. Thus, the LOD is

$$\text{LOD (IADN)} = C_b + 3 \text{ X } S$$

*Representativeness*

This expresses the degree to which data accurately and precisely represent characteristics of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness with respect to the field sampling is a measure of the parameter variation at a sampling point and is evaluated by collecting random duplicate samples (FD1/FD2). Representativeness in IADN also expresses the degree to which the selected monitoring stations reflect loadings conditions throughout the Great Lakes basin and reflects spatial variability among the selected IADN stations.

*Comparability*

Comparability addresses the confidence with which one data set can be compared to another. The comparability of one year's data with another is maintained by adherence to standard operating procedures. Any changes to sampling or analytical procedures are thoroughly evaluated and documented. Comparability between laboratories and projects may be assessed through the analysis of a series of blind PE samples. Field comparability is addressed through implementation of a training program and adherence to SOPs.

Analyte Identification

Samples analyzed by GC-micro ECD, PCB congeners and pesticides, are identified according to their retention time, with determinations made by comparison to the calibration standard. Peaks must be within 0.25-0.5% of the retention time in the calibration standard to be considered a correct identification. For pesticide analysis, all samples are verified for correct identification by retention time on a second column (1701). PAHs and PBDEs are identified and analyzed by GCMS by running in the selected ion monitoring (SIM) mode.

**Table 3.1.** Data Quality Objectives for Trace Organic Compounds

| QA Criteria | QC Code | Sample Type | Frequency | Target | Control Action | Unit |
|---|---|---|---|---|---|---|
| precision | FD1/ FD2 | field: co-located samplers | 5 % | Total PCBs, each pesticide , PBDE, and each PAH, RPD <100% | re-analyze if sample available | % |
| | LD1/ LD2 | laboratory: replicate analyses | 5 % | Total PCBs, PBDE, each pesticide and PAH, RPD <50% | re-analyze same or alternate sample | % |
| accuracy | LSS | Surrogate spikes | all samples | 50% < R < 130% | | % |
| | LMS | Matrix spikes | 1 per 2 batches (5%) | 50% < R < 130% for 70% of individual congeners and for average | Investigate and comment on sources of loss or contamination for both criteria. | % |
| blanks | FB | field matrix blank | 5 % (1/month) | < 20% of associated sample mass | find source of contamination | mass |
| | LB | lab matrix blank | 5% | < LOD | run second LB; eliminate source of imprecision; | mass |
| completeness | | field samples | | 90% | no action; % reported | % |
| calibration | CLM | multiple point calibration 4 point | annual | $r^2 > 0.95$ | re-optimize instrument, repeat calibration | |
| | CCS | Common calibration std | 2/batch (10-12 samples) | | replace calibration daily and regenerate response factors | |
| | CRS | Common Reference Standard | 1/batch | Each compound $\pm$ 20 % of actual mass | Check standard and GC cleanliness | % |
| | LCB | lab calibration blank | 2/GC run | < LOD | check for contamination; re-optimize instrument | |
| Pesticide analysis | | 1701 column confirmation | Each vapor precipitation, and urban filter sample | Minimum values taken (DB-5 vs DB-1701 columns) | | |
| detection limits | | IDL | 1/year | | report in yearly QA Report | |
| detectability | RFS | routine field samples | all samples | > LOD | | |
| holding time | RFS | routine field sample | all samples | 8 months | | |

Please note the reduction in the number of QC samples in MQO in Table 3.1. After generating reproducible results for laboratory blanks and matrix spike for one year, IU requested that the GLNPO Project Officer, Angela Bandemehr, allow us to cut down the number of matrix spike and laboratory blank experiments from 10% to 5%. This was approved on July 23, 1996.

After starting the operation of satellite sites, the number of routine samples increased by 30%. To minimize the cost and to cope up with increased number of samples, IU requested that the EPA allow us to reduce the number of field duplicates from 20% to 10% and laboratory duplicates from 10% to 5%. Angela Bandemehr approved this request on July 23, 1996. The documents are attached in Appendix 17.7. The percent of field blanks and field duplicates were further reduced from 10 to 5% in July 2006 with the permission by Melissa Hulting.

The "Mullin 94" standard (Mullin, M. D., 1985: PCB Workshop) was replaced by a custom made Common Calibration Standard in January 2005. This standard was first distributed by Peter Fowlie in 2004 and is used by all participating laboratories. Later on, Richard Park sent another batch in 2009. The use of a performance standard was stopped on December 2005 because one Calibration Reference Standard was introduced by Canadian QC officer Peter Fowlie on 2001 (See appendix 17.12). The last Canadian QA officer, Helena Dryfhout-Clark, has distributed Common Reference Standards for all target compounds over the last several years. Indiana University will start buying directly Calibration Reference Standards starting from 2018.

**Table 3.2.** Measurement Quality Objectives for Meteorological and Ancillary Parameters

| Parameter | Sample Type | Frequency | Targets |
|---|---|---|---|
| Wind Speed | factory calibration | Annually | |
| Precision | | | ±5% (1-100 mph) |
| Accuracy | | | ±5% (1-100 mph) |
| Completeness | | | 95% |
| Wind Direction | compass reading | Annually | |
| Precision | | | ±5° |
| Accuracy | | | ±10° |
| Completeness | | | 95% |
| Solar Radiation | factory calibration | Annually | |
| Precision | | | ±5% |
| Accuracy | | | ±5% |
| Completeness | | | 95% |
| Temperature | factory calibration | Annually | |
| Precision | | | ±0.5 ° |
| Accuracy | | | ±0.5 ° |
| Completeness | | | 95% |
| Relative Humidity | factory calibration | Annually | |
| Precision | | | ±5% (12-95% RH) |
| Accuracy | | | ±5% (12-95% RH) |
| Completeness | | | 95% |
| Air Sample Volume | data review | | |
| Precision | | 12 days (organics) | ±10% |
| Accuracy | | annually | |
| Completeness | | | 95% |

na = not applicable

**Table 3.3.** List of Target Analytes in IADN samples.

| **PCBs from 2017** | |
|---|---|
| 4+10 | 101 |
| 7+9 | 99 |
| 6 | 119 |
| 8+5 | 83 |
| 19 | 97 |
| 12 | 81 |
| 13 | 87 |
| 18 | 85 |
| 15+17 | 77 |
| 16 | 110 |
| 32 | 135+144 |
| 26 | 123 |
| 31 | 149 |
| 28 | 118 |
| 33 | 114 |
| 53 | 131 |
| 22 | 132+153+105 |
| 45 | 163+138 |
| 52 | 126 |
| 49 | 128 |
| 47 | 167 |
| 48 | 174 |
| 37 | 202+171 |
| 42 | 156 |
| 41+71 | 172 |
| 64 | 180 |
| 100 | 199 |
| 74 | 169 |
| 70+76 | 170+190 |
| 66 | 201 |
| 95 | 207 |
| 91 | 194 |
| 56+60 | 205 |
| 92+84 | 206 |
| 89 | |

| **Pesticides from 2017** |
|---|
| HCB |
| alpha-HCH |
| beta-HCH |
| gamma-HCH |
| heptachlor epoxide |
| alpha-chlordane |
| gamma-chlordane |
| oxychlordane |
| *trans*-nonachlor |
| endosulfan I |
| endosulfan II |
| endosulfan sulfate |
| p,p'-DDT |
| p,p'-DDE |
| p,p'-DDD |
| o,p'-DDT |
| o,p'-DDD |
| aldrin |
| endrin |
| dieldrin |
| |
| **PAHs** |
| fluorene |
| phenanthrene |
| anthracene |
| fluoranthene |
| pyrene |
| retene |
| benz[*a*]anthracene |
| chrysene + triphenylene |
| benzo[*b*]fluoranthene |
| benzo[*k*]fluoranthene |
| benzo[*e*]pyrene |
| benzo[*a*]pyrene |
| indeno[1,2,3-*cd*]pyrene |
| dibenz[*a*,*h*]anthracene |
| benzo[*ghi*]perylene |
| coronene |

| **PBDEs from 2017** |
|---|
| 15 |
| 17 |
| 28 |
| 47 |
| 49 |
| 66 |
| 85 |
| 99 |
| 100 |
| 139 |
| 140 |
| 153 |
| 154 + BB-153 |
| 183 |
| 197 |
| 201 |
| 203 |
| 206 |
| 207 |
| 208 |
| 209 |
| |
| **Other flame retardants** |
| |
| TBE |
| DP-1 |
| DP-2 |
| EHTBB |
| BEHTBP |
| PBBZ |
| pTBX |
| PBEB |
| HBB |
| DBDPE |
| **Others** |
| Total suspended parti- |
| |
| **Meteorological** |
| Temperature |
| Wind speed |
| Wind direction |
| Solar radiation |
| Relative humidity |
| Barometric pressure |

Please note, that analyte list in Table 3.3. was changed following adjustments made in summer 2017 (see appendix 17.20). Specifically, we will stop reporting methoxychlor, PCB-44, BDEs 7, 10, 30, 71, 119, 126, 138, 156+169, 180, 184, 191, 196, 204, and 205, as well as HBCD starting with samples collected after 1 January 2017.

## 4. SITE SELECTION AND SAMPLING PROCEDURES

IU is currently collecting samples at three Master sites and two Satellite sites. Master sites are Eagle Harbor, Michigan, on Lake Superior; Sturgeon Point, New York on Lake Erie; and Sleeping Bear Dunes National Lakeshore on Lake Michigan. The Satellite sites are Chicago, Illinois on Lake Michigan and Cleveland, Ohio, on Lake Erie. An additional QC site at Point Petre, Ontario, was established on Lake Ontario for data comparison with the Canadians in 1998. Site selection criteria had been prepared for this network (Section 17.1) and were agreed to by all participants in IADN in June 1990. Since the goal of the project is to measure regional background concentrations of target compounds, sites removed from local sources and representative of regional conditions were selected. Future satellite sites will meet these site selection criteria as closely as possible, unless intervening research indicates other needs.

Three types of samples are collected in this project: precipitation (rain and snow), airborne particles, and airborne organic vapors. The detail of the current sampling procedures is given in the Field SOP, Collection of Air and Precipitation Samples, version 1.4, May 2010. (Salamova and Bays, 2013)

Precipitation sampling is carried out using equipment and methods developed in Canada (Strachan and Huneault, 1984). The sampler is an MIC Type B precipitation collector modified to provide a heated enclosure for the absorption column. This enclosure is held at 15 °C. Enough heat is transferred to the collection surface so that snow melts on contact. For sample collection, the operator connects a pre-cleaned glass column containing 10 g of clean XAD-2 resin. During a precipitation event, the sampler opens automatically and collected water passes through the column to a receiving flask. After the sampling period, the operator measures the volume of collected precipitation.

Airborne particles are sampled by methods described in sections 2.2.3 and 2.2.4 in the EPA reference manual (U.S. EPA, 1983). The samplers used are Andersen or Tisch TE 300-313 mass flow-controlled, high volume samplers (Model UV-1). These samplers are also fitted with retractable filter covers to prevent passive loading (Sample-Saver, Gen Metal Works). Particle filters are binder-free, quartz fiber filters heated to 450° C prior to use. For vapor collection, the

samplers are modified so that the filtered air passes through a cartridge containing 40 g of XAD-2 resin. Table 4.1 summarizes containers, preservation, and holding requirements for samples that are returned to the IU lab for analysis.

All filters are weighed at the IU lab before analysis or shipment to other laboratories. The filters are all equilibrated at 50% relative humidity (saturated solution of $LiNO_3$) at 25 °C for 24 hours before weighing. The high-volume sampler filters are weighed using a top-loading analytical balance (Mettler, AE50, fitted with a tray for filter) with internal calibration. A reference weight is analyzed, and the weight recorded prior to each weighing session as a quality control measure.

Current sampling schedules and analyses are shown in Tables 4.2. A project schedule is shown in Figure 1.1. The IADN master sites have duplicate precipitation and air samplers for collecting replicate samples and field blanks. These QA samples are collected using the same procedures as the regular samples. The field blanks are collected with the samplers turned off so that no precipitation or air passes over the samples. A set of current sampling procedures is attached (Section 17.2) with a description and examples of the sample coding system.

**Table 4.1.** Sample Containment, Preservation and Holding Times

| Sample Type | Container* | Volume | Preservation | Sample Collection Interval | Maximum Holding Time |
|---|---|---|---|---|---|
| **Precipitation (XAD Resin for organics)** | | | | | |
| Shipment[a] | Glass column | 25 mL | None | 1 month** | 7 days (3 months for winter samples at Eagle Harbor) |
| Storage[b] | Glass jar | 200 mL | -20° | | 8 months |
| **Hi-Vol (XAD Resin)** | | | | | |
| Shipment | Stainless Steel cartridge | 175 mL | None | 12 days | 7 days |
| Storage | Glass jar | 200 mL | -20° C | | 8 months |
| **Filters** | | | | | |
| Shipment | Aluminum foil | | None | 12 days | 7 days |
| Storage | Polyethylene bag | | -20° C | | 8 months |

*All bottle, jar and column closures are Teflon or muffled aluminum foil
** Sampling collection time for precipitation was changed from 28 days to 1 month from August 2004.
[a]Shipment = maximum time between collection and shipment (shipping time = 2 days, held in the field up to 5 days)
[b]Storage = maximum time between field collection and extraction in the laboratory

**Table 4.2.** Annual Sample Collection and Analysis at IADN Sites

| TYPE OF SAMPLES | SAMPLING FREQUENCY | ANALYSIS | NUMBER OF SAMPLES COLLECTED PER YEAR | | | | |
|---|---|---|---|---|---|---|---|
| | | | MASTER (ROUTINE) | SATELLITE (ROUTINE) | QC SAMPLE (POINT PETRE) | DUPLICATE / BLANK % | TOTAL |
| Organics in Precipitation | 1 month | PCBs, PAH, pesticides, and PBDE | 36 | 24 | 12 | 5/5 | 80 |
| Organics in Filter | 12 Days | PAHs (all), PBDE (all) pesticides (selected) | 90 | 60 | 10 | 5/5 | 176 |
| Organics in Vapor | 12 Days | PCBs, PAHs pesticides, and PBDE (all) | 90 | 60 | 10 | 5/5 | 176 |
| **Total Samples for collection** | | | | | | | **432** |
| **Samples for Analysis**<br>**Lab Duplicates**<br>**Lab Blank**<br>**Matrix Spike** | | | | | | **5**<br>**5**<br>**5** | **432**<br>**22**<br>**22**<br>**22** |
| **Total Sample for Analysis** | | | **216** | **144** | **32** | **5%** | **498** |

## 5. SAMPLE CUSTODY AND ARCHIVING

Pre-weighed filters and sample cartridges are prepared and assembled at IU. They are mailed to the site operators who are responsible for installation, retrieval and filling out the field data sheet (Section 17.2). These forms serve as sample custody forms.

All samples are tagged with a unique code number that indicates site, date and sample type. All site activities are logged into a permanent logbook at the site.

After collection and storage at the site, samples from the U.S. sites and associated field data sheets are shipped by Federal Express to IU. The Field Technician reviews the IADN Field Report Forms and confirms the contents by dating and initialing the form. These forms are permanently filed. All XAD-2, and filter samples are stored at approximately –20 °C until extraction and analysis. The maximum holding times are listed in Table 4.1. The project samples and meta-data are under the custody of the Principal Investigator; the Field Technician is the IU Sampling Coordinator. All field data sheets, results of all laboratory analyses, and copies of all site log entries are under the control of the Principal Investigator.

If sample integrity is questionable, the Project Director together with Data Manager decide whether to discard the sample or to analyze it and include it in the database with an appropriate comment. For example if the rain columns are broken, if the filters are torn or wet, or if the XAD cartridges are not well sealed, they are discarded. Unused samples are stored for at least 5 years at -20 $^{0}$C.

## 6. CALIBRATION PROCEDURES AND FREQUENCY

The procedures and the frequency of calibration vary depending on the chemical and physical parameters being determined. The anemometer will be returned to the factory for recalibration. The wind direction sensors are returned to the supplier for maintenance and recalibration. The chip for the relative humidity sensor is replaced once in 2 years. Meteorological sensors are calibrated with a handheld weather monitor three times per year to confirm accurate readings and continuously monitored by site operators for malfunctions. Flow rates for the high-volume samplers are calibrated 3 times a year using EPA procedures (section 2.2.2, U.S. EPA, 1983) with a standard manometer calibrator (Tisch Environmental model TE-5028A Sierra-Andersen Model 335) using a 5 point calibration by the IU Field Technician. Information on the calibrations of field instruments is summarized in Table 6.1. The Hi-vol orifice calibrator is recertified once a year by the manufacturer Tisch Environmental.

Gas chromatographs used for quantification of target compounds are calibrated immediately prior to each run. The PCB calibration standard is prepared from the custom made IADN Standard (solutions from AccuStandard. The source, lot number, identification, and purity of each reference material will be recorded. Reference solutions are diluted using Class A volumetric glassware. Individual stock standards for each analyte are prepared. Combination working standards are prepared by volumetric dilution of the stock standards. The calibration standards are stored at –20° C. Newly prepared standards are compared with the existing standards prior to their use. All solvents utilized are commercially available, distilled in glass, pesticide residue grade and judged suitable for trace organic analysis. Working standards are prepared once every two years. Stock standards will be prepared once per five years. Three custom made common reference standards (CRS) for PCBs, pesticides, PAHs, and PBDEs were supplied by the QA officer from Environment and Climate Change Canada. They will be purchased by Indiana University laboratory starting from 2018. Each is run with every set of samples to confirm the instrument calibration.

PCBs are analyzed by gas chromatography using an Agilent 6890 instrument with electronic pressure control and a micro electron capture detector (GC-μECD) and reported as individual congeners. Individual congeners are identified by retention time (RT) match against the standard. Relative response factors are calculated by dividing these amounts by

their corresponding peak areas utilizing internal standard calibration formulas supplied with the instrument software. Internal standards (PCB congeners 30 and 204) are used for quantitation.

Pesticides are analyzed by gas chromatography on an Agilent 6890 instrument with electronic pressure control and μECD. Calibration is performed using a mixed pesticide standard (Ultra Scientific) by the internal standard calibration method similar to that described above. PCB congeners 65 and 155 are the internal standards for pesticides. Pesticides are analyzed on DB-5 and DB-1701 columns, and the minimum values of the DB-5 vs. the DB-1701 measurements are reported.

PAHs and PBDEs are analyzed by gas chromatographic mass spectrometry (GC/MS) on an Agilent 6890 GC coupled to an Agilent 5973 mass spectrometer and Agilent 7890 GC coupled with Agilent 5975 MS ) in the selected ion monitoring mode (SIM). A mixed PAH standard (**PM-611-1,** Ultra Scientific) and a mixed PBDE standard (BFR-PAR, Wellington Laboratories) are utilized for calibration. The mass spectrometer tune settings are optimized prior to each calibration run. Information on the calibration of analytical instruments is summarized in Table 6.2.

Sampling and analytical logbooks are kept to record inspections, calibrations, standard identification numbers, the results of calibrations, and corrective action taken. Equipment logs for the GC-ECD and GC-MS instruments document instrument usage, maintenance, repair, and performance checks. Daily calibration data will be stored with the raw sample data.

**Table 6.1.** Field Instrument Calibration

| Parameter | Sampling Instrument | Calibration Method | Frequency | Corrective Action |
|---|---|---|---|---|
| Organics (PBDE, PCB/PAH/OCs) | Hi-vol-Anderson (Mod. UV-1) | Anderson orifice calibration | 3 times/year* | adjust flow |
| Wind Speed | Met one: Cup anemometer: model # 024A | Met one | Biennial | Na |
| Wind Direction | Met one: Wind Vane Potentiometer Model # 014A | Met one | Biennial | Na |
| Solar Radiation | Li Cor: Pyranometer Model # L1200S | Li Cor | Biennial | Na |
| Temperature/ Relative Humidity | Vaisala Inc: Probe Model # HMP45A | Vaisala | Biennial | Na |
| Barometric Pressure | Vaisala Inc Model # CS-105 | Vaisala | If necessary | Na |

The equipment calibration frequency (5 point calibration) was changed from 4 times a year to 3 times a year by IU Field Technician. IU requested this change because there was not much work at the site during winter. The calibration shift was also within acceptable limit on quarterly basis. This request was approved in December 1998. Currently the equipment is calibrated in Spring, Summer, and Fall. The documents of request and approval are attached in Appendix 17.8. In between two 5 point calibrations, the local site operator calibrates the sampler using a single point calibration.

**Table 6.2.** Analytical Instrument Calibration

| Parameter | Analytical Instrument | Calibration Method | Frequency | Corrective Action |
|---|---|---|---|---|
| PCBs | GC HP 6890, EPC and micro ECD | ISTD | 2 standards/sample set | Recalibrate daily |
| Organochlorine pesticides | GC HP 6890, EPC and micro ECD | ISTD | 2 standards/sample set | Recalibrate daily |
| PAHs | GC-MS, HP 6890, HP-MS 5973 | ISTD | 2 standards/sample set | Recalibrate daily |
| PBDEs | GC-MS, HP 7890, HP-MS 5975 | ISTD | 2 standards/sample set | Recalibrate daily |

## 7. ANALYTICAL PROCEDURES

Flow sheets of methods used in our laboratory for sample preparation are given in Section 17.3. Method descriptions for sample preparation and instrumental analysis are detailed in IU IADN SOPs (Salamova and Arnold, 2015; Liu et al., 2016). The latest versions are all listed in Section 15'.

## 8. DATA REDUCTION, VALIDATION AND REPORTING

The initial reduction of the analytical data is done with the data system of the GC-ECD or the GC-MS. Identifications of the peaks are done by GC retention times compared to calibration standards. Quantitation is done by the internal standard method. When GC-MS data are used, a primary ion is used for quantitation and a secondary ion is used for confirmation. Once identified, the peak response factor from the calibration table is used to determine the amount of each compound of interest (COI) present. The quantitation internal standards corrects for dilution, detector response and other systematic quantitation variables. Surrogate standards (PCBs 14, 65, and 166) are used to estimate recoveries of PCB congeners in each sample. Recoveries of the organochlorine pesticides are determined by using dibutylchlorendate (DBC) and δ- and ε--HCHs as surrogate standards. Recoveries of PAHs are determined by using $d_{10}$-phenanthrene and $d_{10}$-pyrene as surrogate standards. Recoveries of each compound per batch are determined by matrix spike experiments. Recoveries of PBDEs are calculated by adding BDE 77, BDE 166, and $^{13}C_{12}$-BDE-209 as surrogate standards.

The relative amount of a COI is equal to the ratio of the GC peak areas of the COI relative to the standard corrected for the relative detector response for the ISTDs, relative response factor (RRF). The absolute amount of the COI then is calculated because the amount of the internal standard (ISTD) is known (it is the relative amount times the known amount of the ISTD). The quantification of target analytes in this project is based on adding a known amount of ISTD to all of the samples. The amount of each COI ($Amt_{COI}$) in each sample is:

$$Amt_{COI} = (Area_{COI})_{sample} \; X \; RRF \; X \; (Amt_{ISTD} / Area_{ISTD})_{sample}$$

$$where: \; RRF = (Amt_{COI} / Area_{COI})_{std} \div (Amt_{ISTD} / AREA_{ISTD})_{std}$$

The concentrations of airborne vapor (cartridge samples), particle-bound analyte, are calculated as follows: Airborne Concentration = $Amt_{COI}$/Air Sample Volume. Similarly, the concentration in precipitation is calculated as follows: Concentration in Precipitation = $Amt_{COI}$/Volume of Precipitation. The concentrations of each PCB, pesticide, PAH, and PBDE are reported in $pg/m^3$ for air samples and in pg/L for precipitation samples. For reporting suite PCBs and total PBDE, some congeners are not included: (*a*) Individual congeners that are above 20% of suite PCBs; (*b*) In IU analyses, PCB congener 44 interferes with other contaminant in DB-5 column, and it has been excluded from our analysis and reporting. (*c*) For PBDEs, total PBDE and total brominated flame retardants (BFR) are both reported.

Meteorological data are reported in SI or equivalent units with the correct number of significant figures. Meteorological outliers are checked for reasonableness and excluded only if they are unreasonable for the area and season or if a sensor malfunction is suspected. Analytical outliers are included in the database unless contamination is noted or an instrument malfunctions. In case of sensor malfunction, the data are collected from close-by meteorological stations and reported as alternative data. The source of data and the URL for the site are given.

Quantitative data from each chromatographic run are used to generate a paper report. This report and a copy of the computer file in network drive are permanently archived by the Laboratory Manager. PCB, pesticide, PAH, and PBDE data are electronically transferred to individual spreadsheet files for different site and different matrix. Sampling information is manually transferred to spreadsheets. These files are used to compile annual data reports to the EPA Project Officer and Data Base Manger.

Analytical data are reviewed and validated by the chemists directly responsible for the individual assays and by their respective laboratory supervisor. Notations concerning laboratory related information, which may impact sample results, are provided to the QA Coordinator with the laboratory data. QC samples are associated with each site and laboratory sample set (Table 3.1). When QC samples do not meet the QC acceptance criteria, either the entire sample set is discarded or reported with comments.

The laboratory turnaround time is 10 months from the last sample collection day. For example, all of the 2015 data were reported by 31 October 31 2016. In the summer 2016, it was decided to stop reporting IADN data to the Canadian Database Manager and send it to a QA coordinator hired by the EPA. This change will be implemented with 2017 IADN data. We will need a written agreement for this and instructions on next steps. All data are stored in an IU network drive, and a back-up is stored in an IU server, which is updated every night. These data are stored for indefinite periods. The hard copies of data analysis are stored in the IADN laboratory for 2 years.

## 9. INTERNAL QUALITY CONTROL CHECKS

The following internal QC checks will normally be made as suggested by MacDougall *et al*., 1980 (see Table 9.1):

1. Duplicate rain and air samplers operate on a regular basis at the Master sites. Samples from these collectors can be considered to be replicates for the entire atmospheric deposition quantification procedure (including meteorological and climatological variability).
2. Field blanks are collected at least once a month at one of the sites (5% of samples)
3. Control charts are used to follow the background levels in field and laboratory blanks, and the recoveries of surrogate and matrix spikes.
4. Alternate set of air samples and rain samples processed (see Section 3) contain a laboratory matrix blank (5% of sample). If contamination is found, its source will be investigated and minimized.
5. Analytical calibration standards are run immediately prior to each set of samples to monitor retention times as well as relative detector response. These responses then are used for qualitative and quantitative identification of the target compounds. Instruments are recalibrated prior to each sample set.
6. Surrogate PCB, pesticide, PBDE, and PAH spikes are added to every sample and blank prior to extraction, permitting recovery to be checked on 100% of the samples. Matrix spike samples, containing the full suite of PCB congeners, all PAHs, pesticides and PBDE, will be included with alternate sets of air samples and rain samples processed. The PCB surrogate standards are PCB congeners 14, 65, and 166. The pesticide surrogate standards are delta-HCH, epsilon-HCH, and dibutylchlorendate. The PBDE surrogate standards are BDE 77, BDE 166, and $^{13}C_{12}$ 209. Average recovery of surrogate PCB congeners, PBDE congeners, PAHs, and pesticides must be within a window of 50% to 130%. In addition, if less than 70% of the PCB, pesticide, or PAH target compounds in the matrix spike are not within this window (50% to 130% recovery), the data set are rejected.
7. Common Reference Standards (CRS): Three sets of custom made reference standards (for PCBs, pesticides, and PAHs) were distributed by the QC Officer Celine Audette and Richard Park in 2013 These standards are run with each group of samples to check the stability of the calibration standard and the instrument condition.
8. Supplies and chemicals are bought from same manufacturing companies. Whenever supplies are received they are visually checked for color and packaging, expiration date. The catalog numbers are matched and the certificates of analysis are filed.

**Table 9.1.** Summary of Internal Quality Control for PCBs, Pesticides, PBDEs, and PAHs

| Frequency | Calibration & Standardization Procedure | Control Limits | Corrective Action |
|---|---|---|---|
| 1/yr | Determine instrument linear dynamic range | Linearity by a least squares fit should not be less than 0.95 | Check standards. Prepare new stock if necessary. Follow instrument troubleshooting procedures. |
| 1/sample set | Analyze a calibration standard for instrument calibration | Should be within ±20% | Recalibrate instrument. Prepare new stock and calibration standards. |
| 1/2 sample set | Analyze a reference standard after instrument calibration | Should be within ±20% | Prepare new calibration or reference standards. Check instrument operation. |
| Prior to every chromatographic run | Inject solvent to evaluate the instrumental background for contamination | | Check for possible contamination. Optimize instrumentation |
| 1/sample set | Analyze field matrix blank | <20% of associated sample mass | Check for possible contamination. Optimize instrumentation |
| 1/ 2 sample sets | Analyze lab matrix blank | Value must be $\leq$ LOD. | Run second blank ASAP. If out of acceptance evaluate sources of contamination or calibration error. |
| every sample | Surrogate spikes | Recoveries 50% to 130% | Comment and investigate sources of loss |
| 1 sample/2 sets | Matrix spike | Average recoveries and 70% of individual recoveries 50% to 130%. | Comment and investigate sources of loss |
| 1 sample/2 sets | Laboratory Duplicate Analysis | RPD should be <50% | Comment and investigate discrepancies |

## 10. PERFORMANCE AND SYSTEM AUDITS

Laboratory checks are conducted by GC-MS analysis of the samples. This information is used to confirm identifications of target organic compounds and to determine the presence of contaminants, which generate a false positive response to the GC-ECD. It is expected that there will be sampling and analytical performance audits with the relevant laboratories in Canada using unknowns supplied by a third party. In addition, it is expected that IU will participate in other interlaboratory comparisons and performance evaluation samples as they become available.

External audits of field operations and the laboratory operations are conducted periodically by the IADN QA/QC Monitor. These audits include independent checks on sampler flow rates. The purpose of the laboratory audit is to check on deviations of the analytical procedures from the SOPs.

Technical system audits (TSAs) are conducted throughout the project. The TSA is a qualitative on-site evaluation of the measurement system. The objectives of the TSA are to verify the existence and evaluate the adequacy of equipment, facilities, supplies, personnel, and procedures that have been documented in this QA project plan. TSAs will be conducted on-site by the U.S. EPA Project Officer at key intervals throughout the project.

## 11. PREVENTIVE MAINTENANCE

Routine maintenance on all equipment is carried out according to the manufacturer's instructions. It is the responsibility of the operator of each piece of equipment to ensure that maintenance schedules are followed and that adequate supplies of commonly used spare parts are on hand to minimize downtime.

Field operators are instructed in the routine cleaning, maintenance, and minor repair of all field samplers and monitoring equipment. Actual maintenance activities are noted in the site logbook. A field technician from IU visits the site 3 times a year to check flow calibrations on the high-volume samplers, to check the operation of the meteorological equipment, and to perform repairs that the site operator could not accomplish.

Laboratory equipment maintenance is documented in a logbook for each instrument. Instrument calibrations are performed as described in the SOPs and at intervals described in Table 6.2.

## 12. CALCULATION OF DATA QUALITY INDICATORS

Overall precision is estimated using "collocated field" duplicate samples (CFD) (Section 3). Relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{C_1 - C_2}{(C_1 + C_2) / 2} X\ 100$$

where $C_1$ and $C_2$ are duplicate observed values.

Laboratory analytical precision is assessed by computing the mean, standard deviation (SD), and percent relative standard deviation for the laboratory surrogate (LSS) and matrix spike (LMS) (Section 3).

Accuracy

A number of methods are used to assess the accuracy of the analytical measurements. CFD samples are analyzed at other laboratories or prepared samples are split and portions analyzed by other participating laboratories. Recovery standards (laboratory surrogate spikes) are added to all samples prior to the extraction step to assess losses in sample preparation. The matrix spikes (with PCB, pesticide, and PAH standards) represent the actual analytical recovery for all target organic compounds. DQOs for accuracy are listed in Table 3.1 and in Section 9. If matrix spike recoveries do not meet these standards, then data from that sample set are rejected. If surrogate spike recoveries do not meet these standards, then that sample is reanalyzed or rejected. Important sampling variables affecting accuracy are the flow rate of the high-volume samplers and the measurement of precipitation volume. Bias is the percentage difference between the nominal flow rate set when the instrument is initially calibrated and the actual flow rate determined when the calibration is checked.

Completeness

Completeness is defined as the percentage of valid data (meets all QA requirements) compared to the total number of samples received intact (Section 3).

## 13. CORRECTIVE ACTION

When the measurement objectives described in Section 3 are not met, the PI has the responsibility of ensuring that proper corrective action is taken. The processing of samples that are affected by the procedure out of control is suspended until the system is back in control. The specific corrective actions depend on the nature of the problem. If the problem is related to a field or laboratory procedure, modified procedures are developed and tested as quickly as possible. For example, if the duplicate air sample volumes do not agree within 10% or if the flow rate changes more than 20% between calibrations, the samplers are replaced and returned to IU for repair.

Other internal QA checks are the recoveries of each surrogate in every sample and the recoveries PCB, pesticides, and PAH in matrix spike experiments. The DQO for analyte recovery is 50-130%. All recoveries outside this range are put in the database with comments. If recoveries are consistently outside this range (three sample sets in a row), sample processing will be stopped and the source of the problem discovered.

The relative percent difference for collocated field duplicate samples (total PCBs, each pesticide and each PAH) must be <100%. If this objective is not met, duplicate samples should be re-extracted and analyzed. If no additional sample is available, the data should be flagged.

Target compounds in procedural blanks that are greater than 10% of typical sample concentrations are considered suspect, and samples run in the same set are the suspect compounds. If contamination is consistent (three or more contaminated procedural blanks in a row), sample processing will be stopped to locate the source of contamination.

External audit results are reported to the appropriate technical staff for review, comments, and corrective action. All corrective actions are documented. A review of audit deficiencies and the corrective actions taken are performed as soon as possible by the PI and the QA Coordinator.

## 14. QUALITY ASSURANCE REPORTS

Quality assurance data are tabulated and summarized by the QA/QC Monitor, Project Director, and Data Manager from Indiana University on an annual basis and made available to the U.S. EPA PO and QAPM. The annual QA results include the results from all QC samples like field blanks, laboratory blanks, field duplicates, laboratory duplicates, surrogate recoveries, matrix spike recoveries, linearity, completeness, and IDL.

## 15. THE DATA VISUALIZATION TOOL

IADN Data Viz (https://iadnviz.iu.edu/) is an online platform for IADN data hosted on IU servers. Users can obtain up-to-date information on spatial and temporal trends of chemical concentrations in air that can be sorted, viewed, analyzed, and interpreted using these on-line tools. These include an information portal, with details about the project and the sampling sites; an analytical tool, with details about techniques and methods applied within IADN; and on-line data visualization tool, where the user will be able to visualize spatial and temporal trends of chemicals measured within IADN. The platform was based on the example of GENASIS (Global ENvironmental ASsessment and Evaluation System), which was created at Recetox, Czech Republic, and is part of the Czech national environmental information system.

**Structure of the site**

1. **About**: Details about the project and the sampling sites. The opening page includes a general description of the project and various related information such as resources, contacts, disclaimer,..).
2. **Sites**: An interactive map of the IADN sampling sites
3. **Trends:** The user can visualize spatial and temporal trends of chemicals measured within IADN. Temporal trends are visualized on a dot plot while spatial trends are visualized on a map. In the attempt to keep the output meaningful, the user will be requested to make some choices.
4. **Data download:** Users can download all data for further analysis. Before the download, the user is prompted to provide general information (i.e. name, affiliation and projected use of the data). The website keeps a record of data type downloaded.

**Data availability:** The website was launched in March 2017 and data up to 2013 were included. New data will be uploaded with an annual frequency, but the data will be embargoed for one year. Data uploaded on the website are not blank or recovery corrected. A detailed description of data availability is given below:

1) **Matrix:** vapor, particles, vapor + particles (calculated only when both phases were measured),
2) **Chemicals*:***
   - *basic OCPs*: α-, β-, γ-HCH, HCB, o,p'-DDT, o,p'-DDD, o,p'-DDE, p,p'-DDT, p,p'-DDD, p,p'-DDE, Σ 3 p,p'-DDTs, Σ 6 DDTs, Σ HCHs;
   - *PCBs*: 5-9, 11-13, 15-19, 22, 26, 28, 31-33, 37, 41, 42, 45, 47-49, 52, 53, 56, 60, 64, 66, 70, 71, 74, 76, 77, 81, 83-85, 87, 89, 91, 92, 95, 97, 99-101, 105, 110, 114, 118, 119, 123, 126, 128, 131, 132, 135, 138, 144, 149, 153, 156, 163, 167, 169-172, 174, 180, 190, 194, 199, 201, 202, 205-207, and ΣPCBs;
   - *OCPs*: Aldrin, dieldrin, endosulfan I and II, endosulfan sulfate, chlordanes, endrin, heptachlors, Σ endos, Σ chlordanes;
   - *PAHs*: anthracene, benz[a]anthracene*, benzo[a]pyrene*, benzo[e]pyrene, benzo[b]fluoranthene*, benzo[ghi]perylene, benzo[j]fluoranthene, benzo[k]fluoranthene*, chrysene*, coronene, dibenz[a,h]anthracene*, fluoranthene, fluorene, indeno[1,2,3-cd]pyrene*, phenanthrene, pyrene, Σ PAHs (*classified as possible human carcinogen by the EPA).

*Note*: the website is setup for the upload of PBDEs and FRs. We have held off on this upload until the issue of how to address blanks levels is addressed.

3) **Sites:** Eagle Harbor, Sleeping Bear Dunes, Sturgeon Point, Cleveland, Chicago.

**Statistics:**

1) Time series analysis: plot of all available data on a log scale. The plot also displays a time trend half-life ($t_{1/2}$), if statistically significant.
2) Visualization of geometric mean for a specific year and specific compound on a map

**Data download**: Data downloads will be available based on 2 levels:

1) Immediate download of the data represented in plot or map

2) Download of larger database upon request (complete a form with name, institution and brief description of use).

**Data Upload:** Data are uploaded on the website via a simple and efficient interface. Microsoft excel spreadsheets containing all the data are loaded on the website. The system provides basic error messages that the user can easily address (i.e. missing date, duplicate columns). The spreadsheets containing the raw data are maintained at IU server, separately from the data uploaded to the website, providing double security. Once the data are uploaded, Indiana University Information Technology Services (UITS) handles backups and storage according to the Memorandum of Understanding (MOU) included as Appendix 17.21 at page 131 (see section 3.2.7). Indiana University will handle all the issues related to data upload.

**Site maintenance and security**: The website was designed by Clayton A. Davis, an IU Ph.D. student at the School of Informatics, Computing and Engineering. Before website release, the website was transferred to University Information Technology Services (UITS), who provides comprehensive support (Operating System support -patching, upgrades, and security-, as well as additional support of Active Directory group/OU/GPO management, file share permission support, IU IT Policy and security alignment reporting and review. A signed copy of the Memorandum of Understanding (MOU) is included as Appendix 17.21 at page 131. UITS as part of the MOU will also guarantee the security of the website. IU has authorized the Department of Homeland Security to perform vulnerability scans as indicated in the DHS Scan Authorization Letter in Appendix 17.22 at page 158.

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# 17. APPENDICES

## 17.1 Site Locations and Site Selection Criteria


**Figure 17.1 IADN sampling sites.**

**Table 17.1 List of all IADN sites with code, population within 25 km radius, and coordinates.**

| Site | Code | Population within 25 km radius | Latitude | Longitude |
|---|---|---|---|---|
| Brule River | B | 3,947 | 46.7101 | 91.4353 |
| Burnt Island | U | 475 | 45.8083 | 82.9506 |
| Chicago | C | 3,895,285 | 41.8343 | 87.6238 |
| Cleveland | L | 1,329,642 | 41.4921 | 81.6785 |
| Eagle Harbor | E | 1,330 | 47.4595 | 88.1491 |
| Point Petre | P | 20,691 | 43.8400 | 77.1556 |
| Sleeping Bear Dunes | S | 24,867 | 44.7611 | 86.0586 |
| Sturgeon Point | T | 132,975 | 42.6928 | 79.0389 |

# IADN Siting Criteria

## INTRODUCTION

The Integrated Atmospheric Deposition Network (IADN) for toxic chemical monitoring and research in the Great Lakes was initiated as a response to the requirements of Annex 15 of the Great Lakes Water Quality Agreement of 1987. The network design was created through the efforts of the Atmospheric Deposition Task Force of the Water Quality Board of the International Joint Commission (Voldner and Eisenreich, 1989).

The following siting criteria were selected from those in a number of existing criteria documents (Table 1), many of which were formulated for acid deposition networks (ASTM,1988; NADP,1988; OME,1985; Tew and Eaton,1986; Vet et. al.,1987). While some of the principles involved in a toxics deposition network are the same (eg. accurate measurement of precipitation amount by type), there are chemical distribution differences for some toxic chemicals that are difficult to quantify at present. For example certain toxic chemicals have high vapor pressures and low water solubilities. For these materials, vapor exchange or dry deposition may be more important than inputs from precipitation. Site characteristics conducive to efficient air and particle sampling rather than precipitation sampling would then be most important.

Estimates of emissions by chemical or by industrial process are available from the USEPA and the Canadian Environmental Protection Service for some chemicals such as the PAHs. Where these exist, they have been considered in the siting criteria. Where usage figures do not exist (for example residential pesticide use), previous experience has been used to provide buffer zones around the sites (Hoff,1989).

All stations in the IADN will be required to submit the site description document (see attached copy) to the Parties and the Annex 15 Working Group for review. Non-compliance with any of the following criteria should be documented to explain of why the criterion could not be met. The Working Group will examine the siting choice in these cases and recommend whether any remedial procedures should be adopted to deal with the non-compliance.

## IADN SITING CRITERIA

1. The primary siting criterion is that the sites be representative of the regional atmospheric environment over the Lakes. All variances from these criteria should be noted on a standard siting checklist and a site map including a 1 km radius around the site. In general only minor deviations from these criteria should be allowed for master sites. More significant deviations are permitted for satellite sites; however, there should be no more than one major source nearby and it should be downwind during prevailing winds.

2. Sites should be at least 40 km from major sources such as larger urban centers (pop.>10,000), heavy industry (chemical plants, foundries, steel mills, smelters, refineries, pulp and paper mills) or other major sources of airborne metals or organic chemicals (large incinerators, power plants emitting more than 10,000 tons/yr SO2, NOX or 100 kg/yr total PAH, major airports, large sewage treatment works).

3. Sites should be at least 10 km from other important sources such as urban areas (pop.=1,000 to 10,000), mining and manufacturing facilities, major highways, commercial areas, electrical transfer stations or smaller sources (lower by a factor of 10) in the categories listed in criterion number 2.

4. Sites should be at least 1 km from local sources such as vehicle or boat traffic (>30 vehicles/hr), farms and tilled fields (in regions where farming is not the predominant land use), fuel or chemical storage areas, landfills, sewage lagoons or small towns (pop.<1,000). The use of pesticides in farming operations within 1 km of the sites should be documented.

5. Sites should be at least 250 m from single residences, parking lots, grazing animals, public roads (<30 vehicles/hr) and other sources in the immediate area of the samplers. Single residences within 1 km of the site should be downwind during prevailing winds or outside of a 22.5° sector on each side of the prevailing wind direction. For all residences within 500 m of the site, type of heating and fuel usage, open burning and usage of household chemicals on the Annex 1 list should be documented.

6. Samplers should be sited on open level terrain (slope<15%) with regionally representative ground cover (preferably grass with a height of no more than 20 cm). The samplers should subtend a vertical angle of less than 30° (2:1 fetch to obstruction height) with any obstruction (trees, towers, power lines). This should be increased to 10:1 for solid structures. In areas with high snowfall, the site should be sheltered by trees at a distance between 2.5 and 4 times their height. The samplers should be on 1 m platforms or no higher than the maximum snowpack.

7. Sites should be no more than 1 km from the lake shore. Where there is a choice, sites should be situated so that prevailing winds are onshore from the lake. Protection by trees from wind and lake spray at a distance from 2.5 to 4 times tree height is also desirable.

8. Sites should have all-weather access, electrical power (200 amp, 125 v) and a security fence if not otherwise secured. Sites should be large enough to allow a 2 m spacing between samplers.

9. No development (industry, construction) should be planned in the vicinity of the site (1km) and the site owner should agree to continuous operation of the site for at least 5 years.

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TABLE 1. REGIONAL SITING CRITERIA FROM OTHER NETWORKS*

| | CAPMON | APIOS | NADP | ASTM | EMSL |
|---|---|---|---|---|---|
| Large Industrial Sources | 50 | 50 | 20 | 10-50 | 10-50 |
| Cities (>10,000) | 40 | | 40 | 10 | 5 |
| Towns (5-10,000) | 10 | | | 10 | 1 |
| Villages (1-5,000) | 5 | | | 1-10 | 1 |
| Airports | | | | | |
| Large | 10 | | | | 2 |
| Small | 3 | | | | |
| Roads (>30 veh./hr) | 0.5 | 1 | | 0.1 | 0.10 |
| Roads (<30 veh./hr) | 0.5 | 1 | 0.1 | 0.1 | 0.05 |
| Small Local Sources: | | | | | |
| construction areas | 0.5 | 1 | | 0.5 | 0.25 |
| residences | 0.5 | | | | |
| parking lots | 0.5 | | 0.1 | | 0.10 |
| service stations | 0.5 | | | | |
| dust sources | 0.5 | | | | |
| fuel or chemical storage | 0.5 | | 0.1 | 0.1 | 0.1 |
| landfills or sewage lagoons | 0.5 | | | | 0.1 |
| farms and feedlots | 0.5 | | 0.5 | 0.5 | 0.5 |
| Distance from Obstructions | 2.5xht | 2.5xht | 2xht | 2xht | 3xht |
| Max. % Slope | | | 15% | 15% | 22% |
| Sampler Height | | | <average snowpack | | <1m |
| Sampler Separation | | | 5m | 2m | 2m |

*Minimum distances in km except where noted otherwise

# IADN SITE DOCUMENTATION CHECK LIST

This check list is intended to provide uniform site documentation for all Integrated Atmospheric Deposition Network sites. It is especially important to list all variances from the siting criteria so that the effects of local sources can be estimated.

## A. General Site Information

1. Site Name:

2. Site Operator:

3. Site Mailing Address:

4. Latitude: Longitude:

5. Provide photographs or slides from the site out toward the 8 compass directions (N,NE,E,SE,S,SW,W,NW).

6. Provide a site map out to 1 km from the site showing trees and other vegetative cover, power lines, roads, buildings and residences, parking lots, tilled fields, swamps, landfills, sewage lagoons, farms, construction sites, fuel or chemical storage areas or any other potential sources of chemical or dust emissions.

7. Provide a base map (1:10000 or the best resolution available) showing features, population and land use in a 10 km radius around the site.

8. Climate

Annual Average precipitation:
Average winter snowpack:

| | January | July |
|---|---|---|
| Average Maximum Temperature: | | |
| Average Minimum Temperature: | | |
| Average Wind Speed: | | |
| Average Precipitation: | | |

Provide a windrose showing annual wind direction frequency from the 8 compass directions at the nearest official weather station.

**B. Regional Information**

For all the categories below, give the distance from the site in km and the compass direction (N,NE,E,SE,S,SW,W or NW). Include all sources in each category that are within 40 km (25 miles) of the site.

1. Urban areas, towns and villages

| Name | Population Winter | Population Summer | Distance | Direction |
|---|---|---|---|---|

2. Large industrial sources such as chemical plants, smelters, foundries, steel mills, refineries, pulp and paper mills, power plants, large incinerators, sewage treatment works, mining and manufacturing.

| Name | Location | Major Air Pollutant(s) | Distance | Direction |
|---|---|---|---|---|

3. Other important sources such as major transportation routes (expressways, main rail lines, shipping routes, commercial airports), chemical or fuel storage depots, electrical transfer stations and any regulated sources of air pollutants.

| Name | Location | Distance | Direction |
|---|---|---|---|

5. For residences within 500 m of the site and farms or commerci- facilities within 1 km of the site indicate fuel type and annual usage (gal , cu. ft., cords), number of days of open burning and annual pesticide use fo Annex 1 chemicals (kg).

| Name | Distance | Direction | Fuel | Open Burning | Pesticides |
|---|---|---|---|---|---|

6. List all other sources of air pollutants such as vehicle traffic on paved or unpaved roads (veh./hr), service stations (kg/yr), fuel and chemical storage (kg/yr), construction sites,landfills, mining and manufacturing within 1 km of the site. Indicate if pesticides or oiling are used alo roads or power lines.

| Source | Distance | Direction | Pollutant(s) | Emission Factor | Pesticide or Oiling |
|---|---|---|---|---|---|

7. Indicate any other problems especially those involving any future (5 to 10 years) changes planned within 1 km of the site or major new sources within 40 km of the site.

D. On-Site Information

8. List materials used for fencing, platforms, buildings, towers and coatings or preservatives. Indicate any other on-site potential sources of air pollutants such as pesticide use, chemical toilets, open burning, generators, fuel or chemical storage, gasoline engines and electrical transformers.

## 17.2. Field Procedure and Field Report forms

INDIANA UNIVERSITY
SCHOOL OF PUBLIC AND ENVIRONMENTAL AFFAIRS
IADN WEEKLY SITE VISIT CHECK LIST

1. Refer to the monthly protocol for specific sampling Dates.

2. Collect samples and measure the volume in the MIC sampler and check the operation of the sampler and the heater. Set up the MIC for the next sampling cycle. Mail XAD-2 columns and a data sheet for each sample to IU laboratory.

3. Check the Campbell data logger for operation. Meteorological data is downloaded once a week from the US sampling sites.

4. Collect filters and cartridges from the organics high volume samplers. Replace the cartridges and filters. Set the timers to operate on the next sampling day specified. Mail filters, cartridges, and a data sheet for each sampler to the IU laboratory. Refer to High Volume procedure sheet for details of sampling.

5. Make an entry in the site logbook and notify IU personnel if there is any problem.

MSB II, room 324
702 North Walnut Grove Ave.
Indiana University
Bloomington
IN 47405

Contact Names and Telephone numbers
**James Bays 812-856-4364**
**Karen Arnold 812-856-2887**
**Amina Salamova 812-855-2926**

Revision No. 7.0
May 2018

**INDIANA UNIVERSITY**
## SCHOOL OF PUBLIC AND ENVIRONMENTAL AFFAIRS

# INSTRUCTION FOR FILTER AND CARTRIDGE CHANGE FOR HIGH VOLUME SAMPLER

1. Install pre-weighed glass fiber filter (labeled side up) in a filter cassette. Do this indoors if possible.

2. If sampler has an exposed filter turns on the sampler. Record the reading on the magnehelic gauge after 2 min. Remove the cassette with the exposed filter. Record the timer reading. Take the cassette indoors and wearing plastic gloves, remove the exposed filter and fold in half lengthwise with the deposit side facing in. Wrap the filter securely in aluminum foil, label and seal in plastic bag.

3. Install the clean filter cassette and tighten the thumbscrews holding the cassette to the sampler inlet.

4. Remove the exposed steel cartridge, wrap it in aluminum foil, and seal in a labeled metal can. Replace a clean cartridge in the holder.

5. Record the timer reading. Test by turning on the hi-vol and allow it to run for 2 min. Record the reading on the magnehelic gauge. Turn off the hi-vol.

6. Set the timer for the desired sampling period. Normally the sampler should start in the morning (9:00 local) and run for 24 hours until the following morning (9:00 local) once every 12 days for organics.

7. Mail the filters and the cartridges to the address below. Copies of the data forms should be sent along with the samples to the address below.

Karen Arnold/James C. Bays
School of Public and Environmental Affairs
Multidisciplinary Science Building, II
702 N. Walnut Grove Avenue
Indiana University
Bloomington, IN 47405

James C. Bays 812-856-4364
Karen Arnold 812-856-2887

8. Sample codes

| Site ID | Sample | Sample Type |
|---|---|---|
| B - Brule River | H - Hi-Vol | 01 - Routine Sample |
| S - Sleeping Bear Dunes | C - XAD Cartridge | 02 - Duplicate |
| T - Sturgeon Point | F - Filter | B - Field Blank |
| E - Eagle Harbor | B - Blank | |
| C - IIT Chicago | | |
| L - Cleveland | | |
| P - Point Petre | | |

Example: SH-OlC-950119 is the code for a routine organics Hi-Vol sample collected at the Sleeping Bear Dunes site on January 19, 1995 (date sample is removed from the sampler)

INDIANA UNIVERSITY
SCHOOL OF PUBLIC AND ENVIRONMENTAL AFFAIRS

## INSTRUCTIONS FOR XAD-2 COLUMN CHANGE WITH MIC SAMPLERS

1. Make sure that all precipitation has passed through the column. If rain or snow has been recent and precipitation is still eluting from the column, wait until all the liquid has drained from the funnel. If the system is plugged, catch any standing liquid in a clean pyrex beaker and pass it through the column.

2. Measure and record the total precipitation volume using the graduated cylinder.

3. Wearing plastic gloves, rinse the Teflon collection surfaces with about 400 mL of deionized water while scrubbing with a piece of glass fiber filter (half of an 8x10" filter with serial no. removed) to remove deposited particles. Allow these rinsings to pass over the column until the water level is halfway between the top of the column and the top of the resin bed. Remove the column; cap both ends with Teflon plugs (make sure the black O-rings are in place). Seal the glass fiber filter in a sample jar; label and package the column and jar for shipment. During the winter (Nov-April), the package should be marked "do not freeze"; during the summer (Jun-Sept), shipment should be in a insulated container with ice packs.

4. Clean the collector surfaces by rinsing with 200 mL of pesticide-free methanol followed by approximately 1 liter of clean tap water with additional scrubbing. Use a test tube brush to clean the funnel outlet. This should be followed by another rinse with 200 mL of deionized water. Discard these rinsings.

5. Install a new column making sure the top and bottom O-rings are in place. After opening the outflow valve and positioning the outflow tubing (see attached diagram), add about 50 mL of deionized water to the collection funnel. Make sure that this water flows through the system; then empty the receiving jug. Wrap the column tightly with aluminum foil to exclude light.

6. Send the column and a data sheet for each sample to:

Amina Salamova/James C. Bays
School of Public and Environmental Affairs
Multidisciplinary Science Building II
702 N. Walnut Grove Avenue
Indiana University
Bloomington, IN 47405

James C. Bays 812-856-4364
Karen Arnold 812-856-2887

9. Sample codes: Site P # sampler -yy-mm-dd where P is precipitation

| Site ID | Sample | Sample Type |
|---|---|---|
| B - Brule River | P = precipitation | 01 - Routine Sample |
| S - Sleeping Bear Dunes | | 02 - Duplicate |
| T - Sturgeon Point | | B - Field Blank |
| E - Eagle Harbor | | |
| C - IIT Chicago | | |
| L - Cleveland | | |
| P - Point Petre | | |

# IADN Field Data Sheet

## Integrated Atmospheric Deposition Network

## Weekly Site Visit Sheet

**Station:** _____ **Operator:** _____ **Received:** _____ **Initial** **Date**

**Calibration Date:** _____ **Date shipped:** _____

Instructions: Fill in all applicable spaces, enter general weather conditions (sunny, raining, etc.) and approximate values for weather variables. Enter OK after Operation for each sampler tested, if the sampler is operating properly; if there is a problem enter "X" and describe the problem at the bottom of the page. For the Hi-Vols, fill in the Timer and Magnehelic readings in the appropriate spaces. For the MICs, enter the temperature inside the sampler and the approximate volume in the carboy. For all samplers, indicate with an "X" whether a sample was collected this week and if the sampler was set up for another run. Indicate with an "OK" if the wind vane is pointing in the proper direction and if the anemometer is turning.

| **Organics Hi-Vol** | Sample ID | Filter ID | Start Date yy-mm-dd | End Date yy-mm-dd | Timer on | Timer off | Magnehelic start | Magnehelic stop |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

**Station:** _____ **Date:** _____ **Time:** _____

| **Precipitation** | Sample ID | Start Date yy-mm-dd | End Date yy-mm-dd | Volume in L |
|---|---|---|---|---|
| | | | | |

Weather _____ Temp _____ Wind dir _____ Wind spd _____ Bar press _____

Organics Hi-Vol #1 Operation _____ Timer _____ Magnehelic _____ Sample: Collected _____ Set up _____

| **Dicot** | Sample ID | Filter ID | | Start Date yy-mm-dd | Timer end | Timer Start | Rotameters | |
|---|---|---|---|---|---|---|---|---|
| | | Coarse | Fine | | | | C | T |
| | | | | | | | | |
| | | | | | | | | |

Organics Hi-Vol #2 Operation _____ Timer _____ Magnehelic _____ Sample: Collected _____ Set up _____

MIC #1 Operation _____ Temp _____ Volume _____ Sample: Collected _____ Set up _____

MIC #2 Operation _____ Temp _____ Volume _____ Sample: Collected _____ Set up _____

| Sample ID | Remarks | Code |
|---|---|---|
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

Met Tower Anemometer _____ Wind Vane _____

Problems and general observations:

Operator _____

**Do not write in the shaded area**

**Field Data form**

## IADN Field Data Sheet

**Station:** _____ **Operator:** _____ **Received:** _____
**Initial** **Date**

**Calibration Date:** _____ **Date shipped:** _____

| **Organics Hi-Vol** | Sample ID | Filter ID | Start Date yy-mm-dd | End Date yy-mm-dd | Timer on | Timer off | Magnehelic start | Magnehelic stop |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

| **Precipitation** | Sample ID | Start Date yy-mm-dd | End Date yy-mm-dd | Volume in L |
|---|---|---|---|---|
| | | | | |
| | | | | |

| Sample ID | Remarks |
|---|---|
| | |
| | |
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| | |
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| | |

**Do not write in the shaded area**

**Field Log Sheet**

*Site Name:* *Site Operator:*

| Date | Arrival | Departure | MIC col installed | MIC col removed | XAD/QF installed | XAD/QF removed | shipped |
|---|---|---|---|---|---|---|---|
| | | | | | | | |
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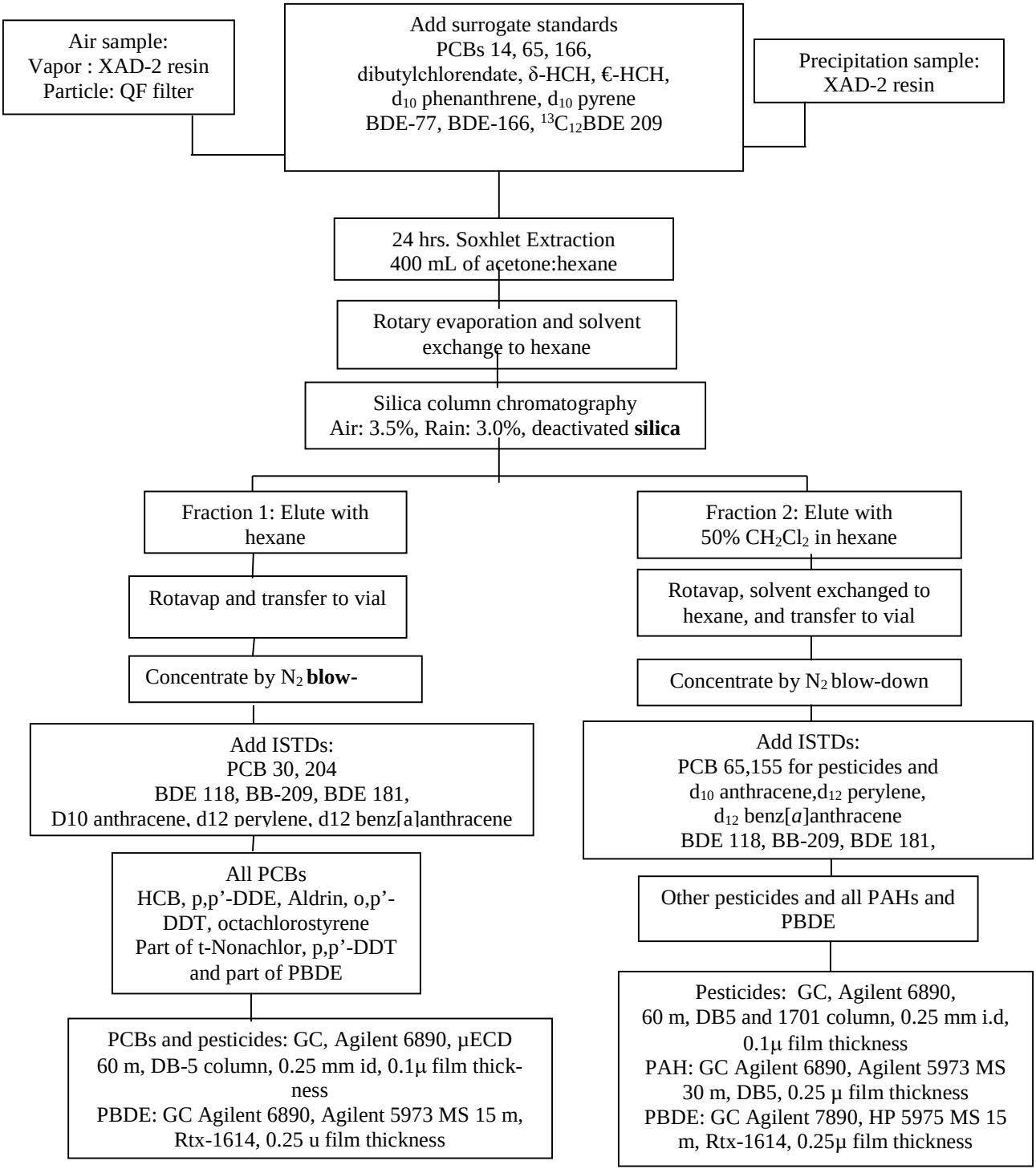
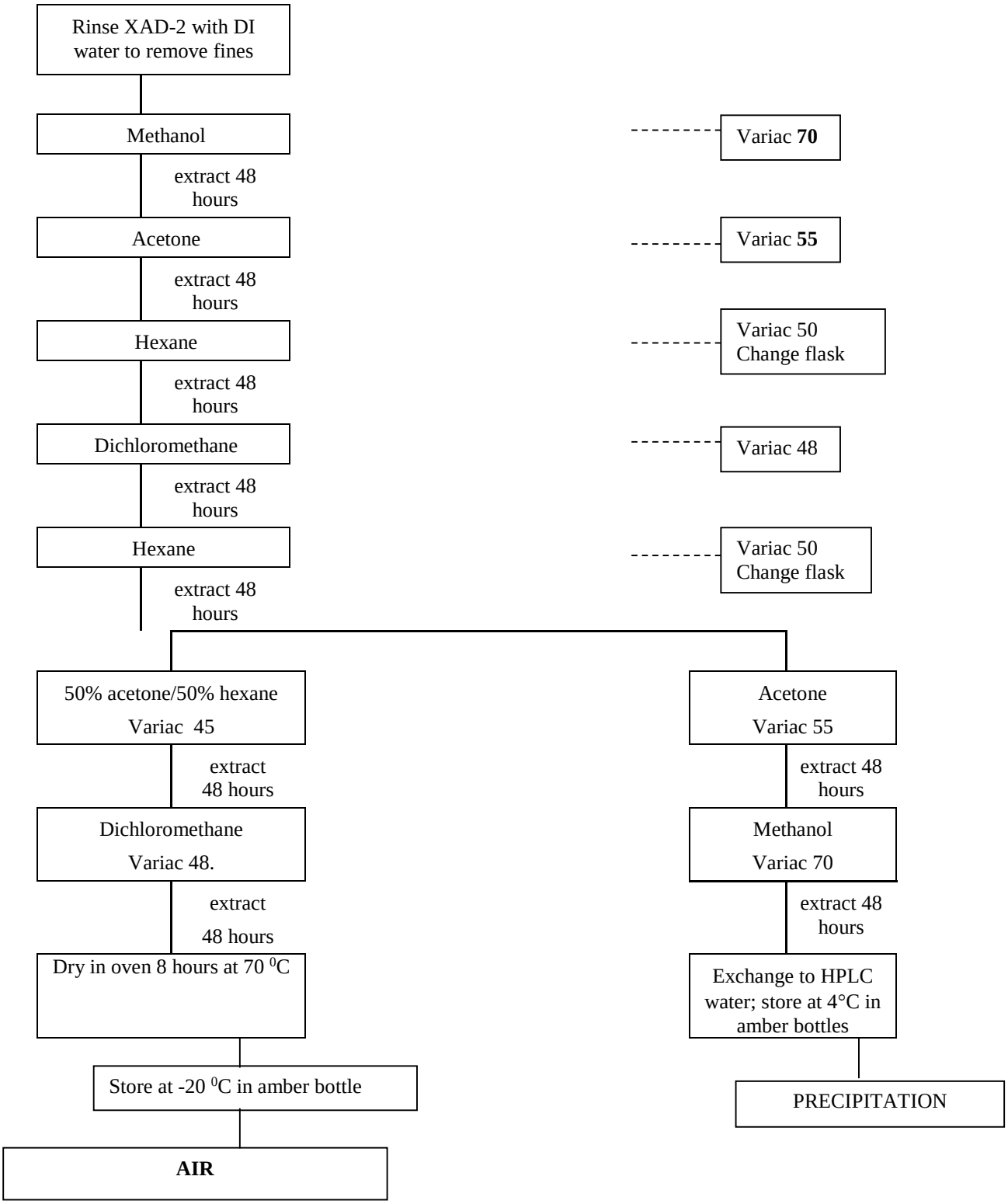
## 17.3. Laboratory Flow Charts

**SUMMARY OF SAMPLE PREPARATION**

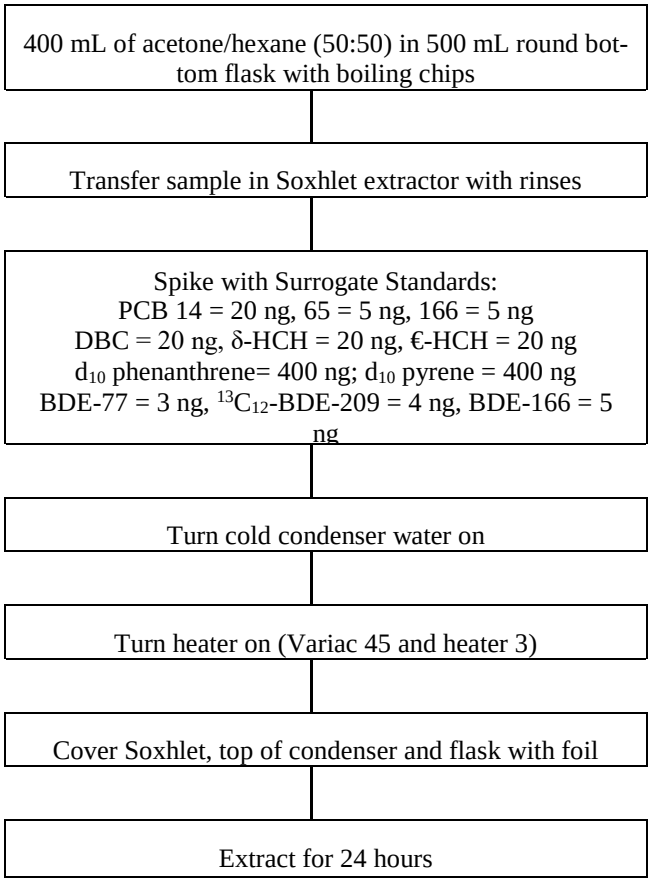


**SUMMARY OF XAD-2 PRE-CLEANING**

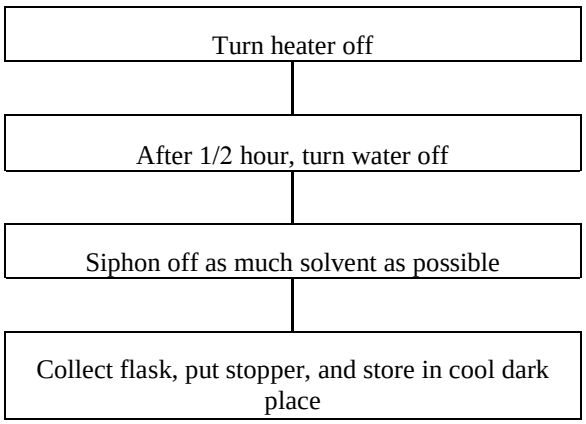


## SUMMARY OF EXTRACTION OF AIR SAMPLES

**Setting up extraction: Day 1**

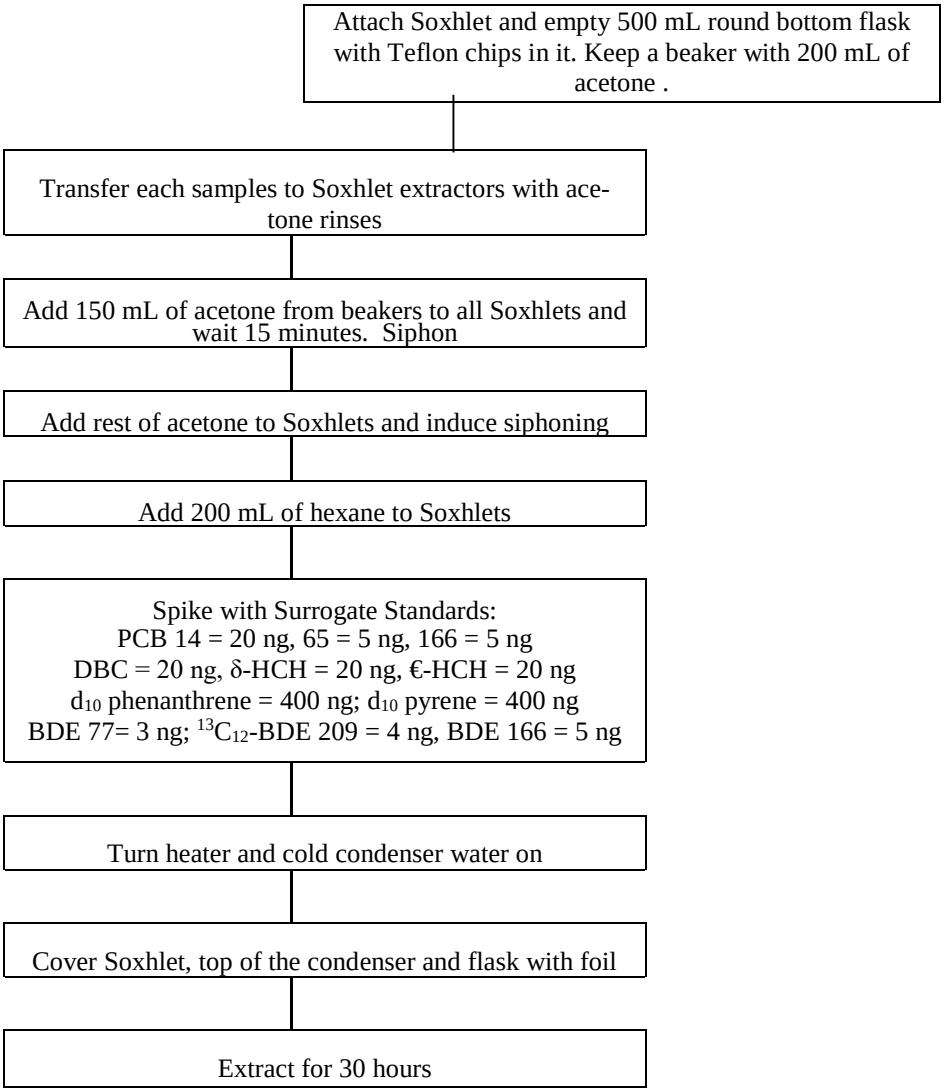



**Taking extraction down: Day 2**

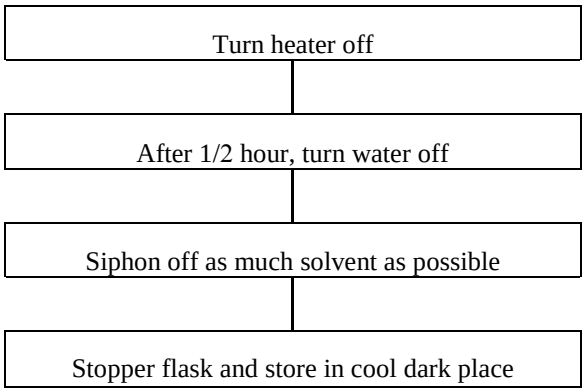


SUMMARY OF EXTRACTION OF PRECIPITATION SAMPLES

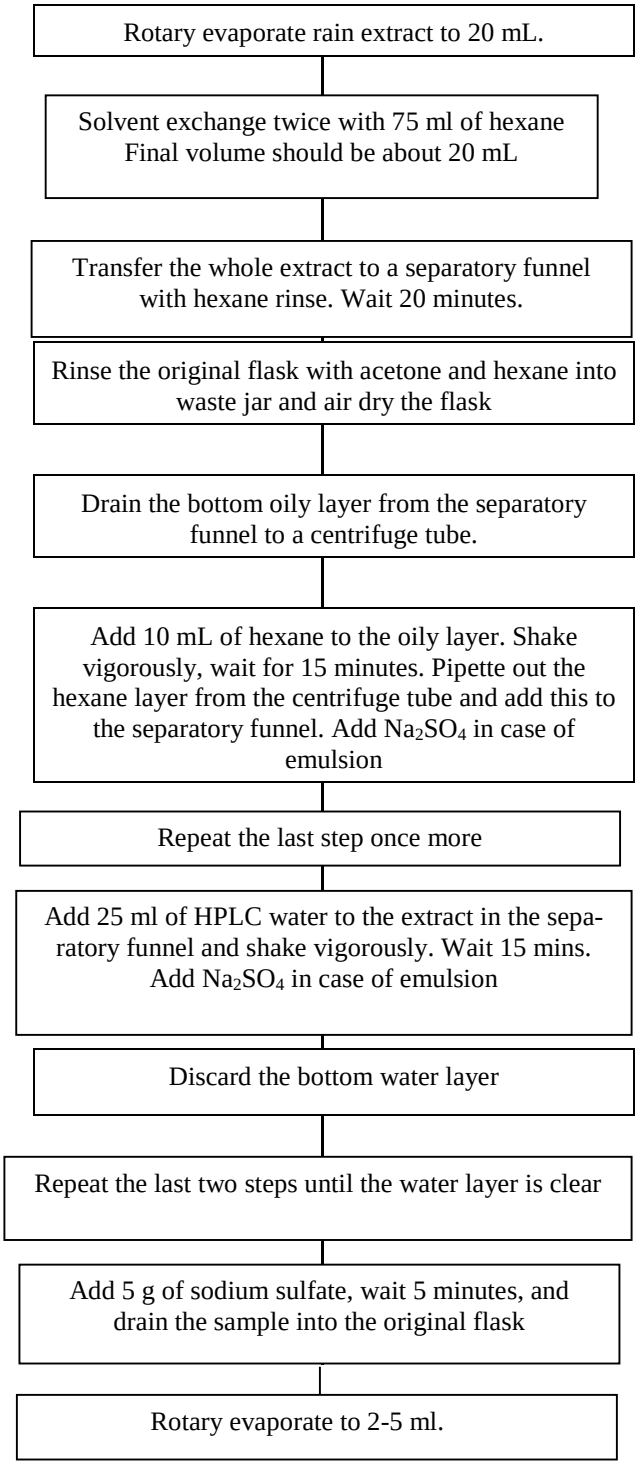
**Setting up extraction: Day 1**



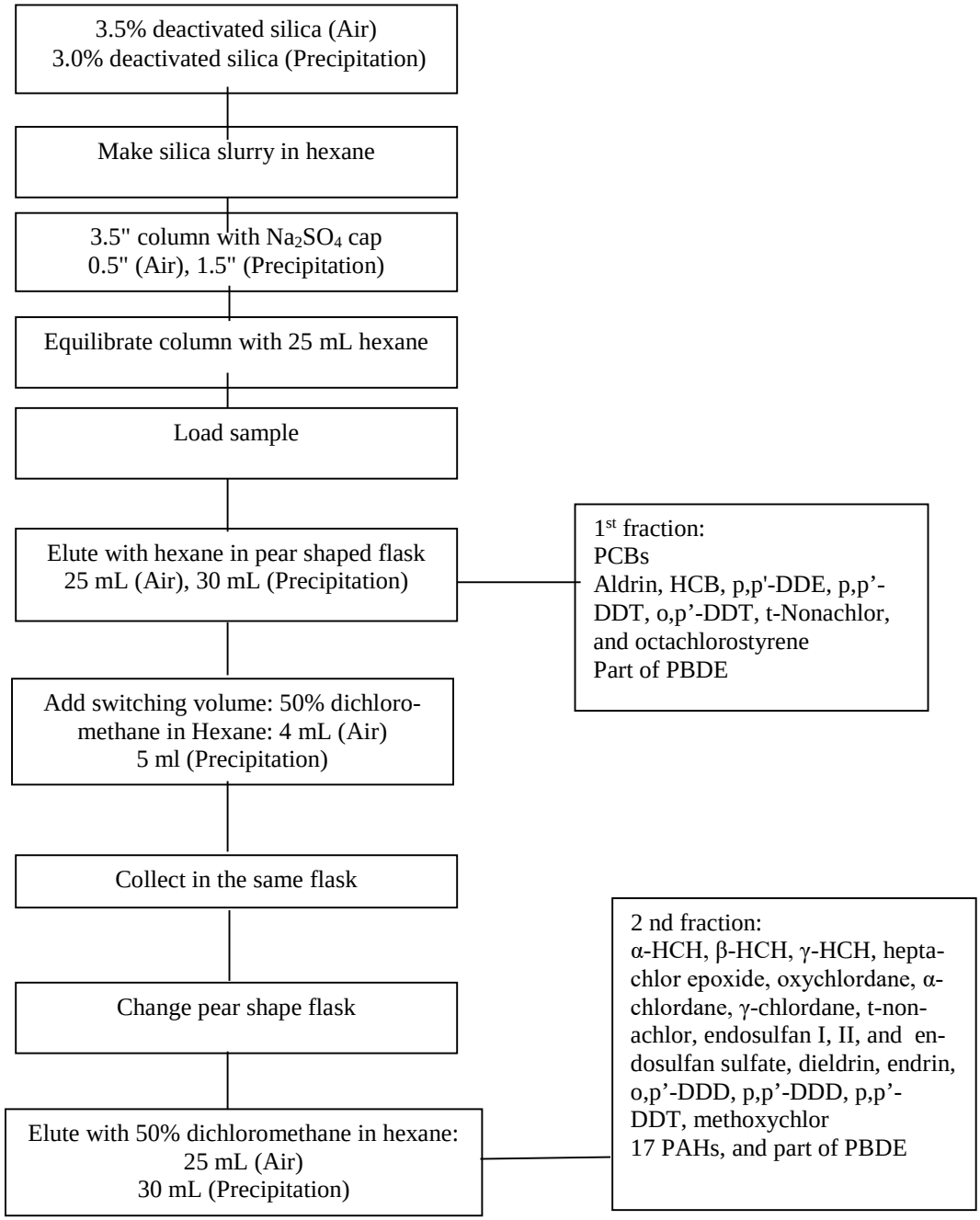

**Taking extraction down: Day 2**



ROTARY EVAPORATION AND BACK EXTRACTION OF PRECIPITATION EXTRACTS



**SUMMARY FOR SILICA COLUMN CHROMATOGRAPHY**



## 17.4. Proposal and approval of stopping TSP/TOC

ANA UNIVERSITY

June 27, 1996

Ms. Angela Bandemehr
United States Environmental Protection Agency
Great Lakes National Program office
77 W. Jackson Boulevard
Chicago, Illinois 60604

SCHOOL OF
PUBLIC AND
NVIRONMENTAL
AFFAIRS

Subject: TSP/TOC in IADN project.

Dear Angela

I am sorry that I missed the IADN meeting at the IAGLR conference. I understand that many action items were discussed. I heard from Ron that there was a discussion about TSP/TOC part of the project. Ron also asked me to look for certain areas where we can reduce the expenses of this project so that we can use that money for the loading study.

One idea is to cut down the number of QA/QC samples. I already sent you a letter about this with some suggestions on June 11, 1996. I am waiting for your approval on that. Secondly, I think that we are spending lot of money and time on TSP/TOC measurements which is giving us little information.

So far, Indiana University has collected TSP/TOC samples for almost 2 years. We have analyzed and processed samples collected from August 6, 1994 to December 30, 1995. Michael Wassouf, an undergraduate student, has worked on TSP/TOC analyses for his class project. I am enclosing the abstract, a table, and a graph from his report showing the seasonal variations. I have also looked at Clyde's

mington, Indiana
47405-2100

recent data from the Illinois State Water Survey from the Winter 1992 to the Winter 1994 (copy attached). Data from these 3 years show the same seasona trends for both TSP and TOC. They are highest at the summer months and low during the winter months. Clyde's data show that, at all three sites, TOC accounted for 11% of TSP whereas the IU data show 14% . Previously, Clyde reported results from the Winter1990 to the Fall 1992 showing a similar trend.

Quartz fiber filters are expensive. We use more than 250 filters every year whi costs about $1500. One student spends at least 500 hours per year which costs about $3500. Maintenance cost on the equipment and shipping costs for the samples are also more since we collect TSP samples more frequently.

In summary, between Illinois and Indiana, we have 5 years' data in hand showi the same trend. I do not see any additional information on TSP/TOC that could be acquired by collection of more samples. Therefore, we request that TSP/TO measurements be dropped from IADN. If you agree, it will save time and money and allow us to focus these resources on the scientific features of IADN.

Sincerely yours
Ilora Basu

(Ilora Basu)
812-855-2926

## 17. 5. Proposal and approval of discontinuing filter composites

United States Environmental Protection Agency
Great Lakes National Program office
77 W, Jackson Boulevard
Chicago, Illinois 60604

Subject: Request to modify the Indiana University IADN Quality Assurance Project Plan

Dear Angela

This letter is a follow-up on our conversation in Niagara Falls about some changes in the IADN sample analysis plan. Ilora and I are requesting that you modify our Quality Control Quality Assurance Plan (QAPP), Revision 3, which was submitted on 7/18/95 and approved on 7/26/95 by Jackie Bode and Michael Papp. The requested changes are as follows:

1. The filters should not be composited. We are loosing at least 50% of the information, especially for the PAHs, by compositing two to three filters. For example, we cannot correlate the composite data with daily meteorological information or with corresponding gas-phase data. With the exception of the PCBs, we cannot just ignore the filters. For example, there is a considerable amount of PAH, especially benzo[*a*]pyrene, on the filter samples. We are not concerned about detection from single filter extracts: We will simply concentrate the extracts to 0.5 mL instead of 1.0 mL; in this way, the detection limit will remain about the same.

2. Analyzing every filter extract, without compositing, will increase our work-load by about 20% (or more) but we propose to compensate for this extra effort by dropping the PCB analyses of the filter samples. We feel this is justified because 90-95% of the atmospheric PCBs are in the gas-phase samples. (Please see the enclosed plots of gas- versus particle-phase PCB concentrations in air samples collected from the 3 master sites from August 1994 to February 1996.) Also, I should point out that Ray Hoff has dropped the analysis of the filters for PCBs at the two Canadian master sites.

3. We are also proposing to stop analyzing PAHs with molecular weights less than 200. These compounds are not health hazards, and they are not environmentally interesting. This change will not save much sample preparation and analysis time, but it will save considerable time during the reporting phase of the project, particularly for QC flags.

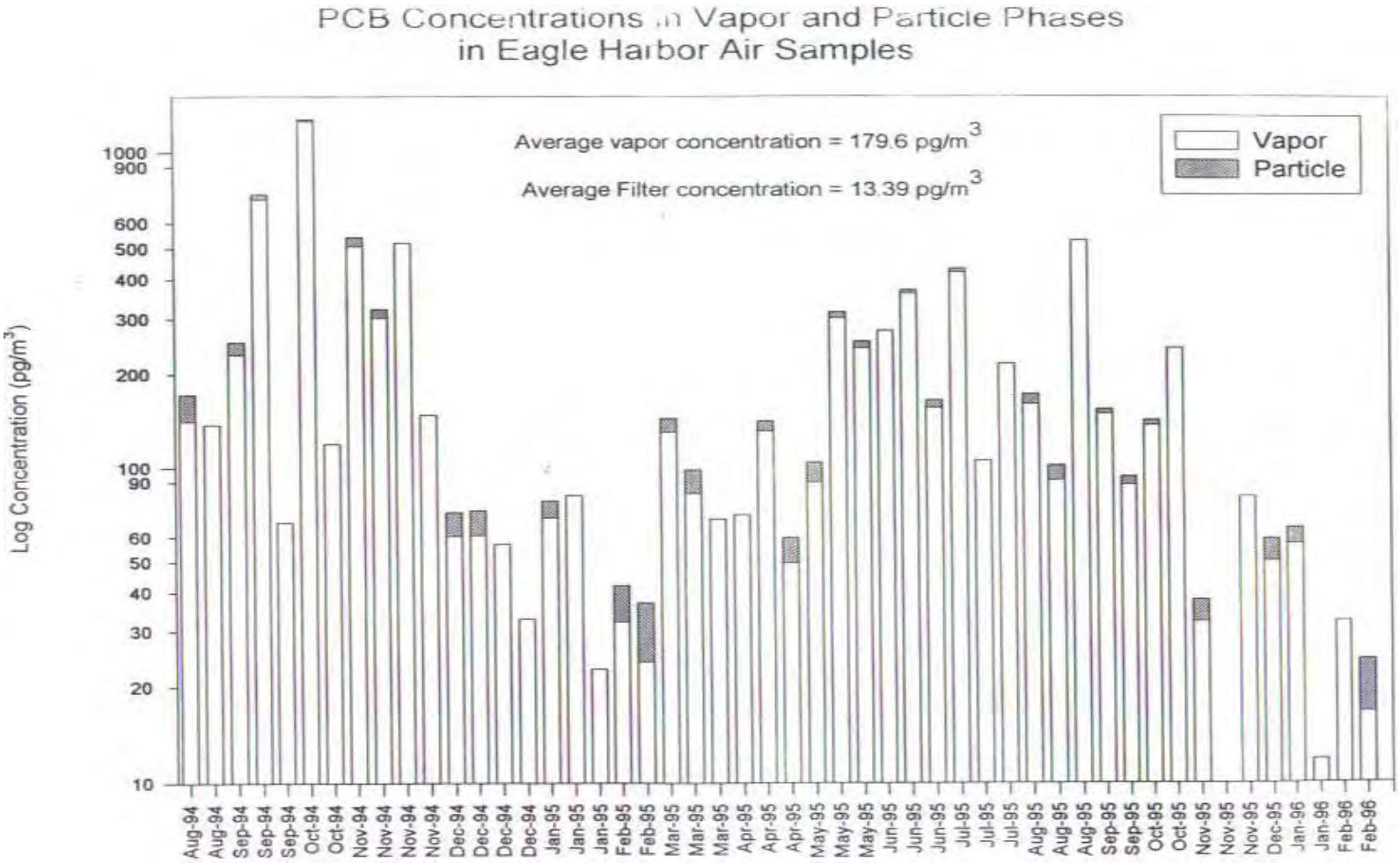
We will be waiting for your approval. In the mean time, we will stop working on filters temporarily until we hear from you.

If you have questions please call me or Ilora at 812-855-2926.

Sincerely yours

Ronald A. Hites
Distinguished Professor
RAH/hs

IADN / angela7.doc


PCB Concentrations in Vapor and Particle Phases
in Eagle Harbor Air Samples
Average vapor concentration = 179.6 pg/m3
Average Filter concentration = 13.39 pg/m3
Vapor
Particle
Log Concentration (pg/m3)
1000
900
600
500
400
300
200
100
90
60
50
40
30
20
10
Aug-94
Aug-94
Sep-94
Sep-94
Sep-94
Oct-94
Oct-94
Nov-94
Nov-94
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Dec-94
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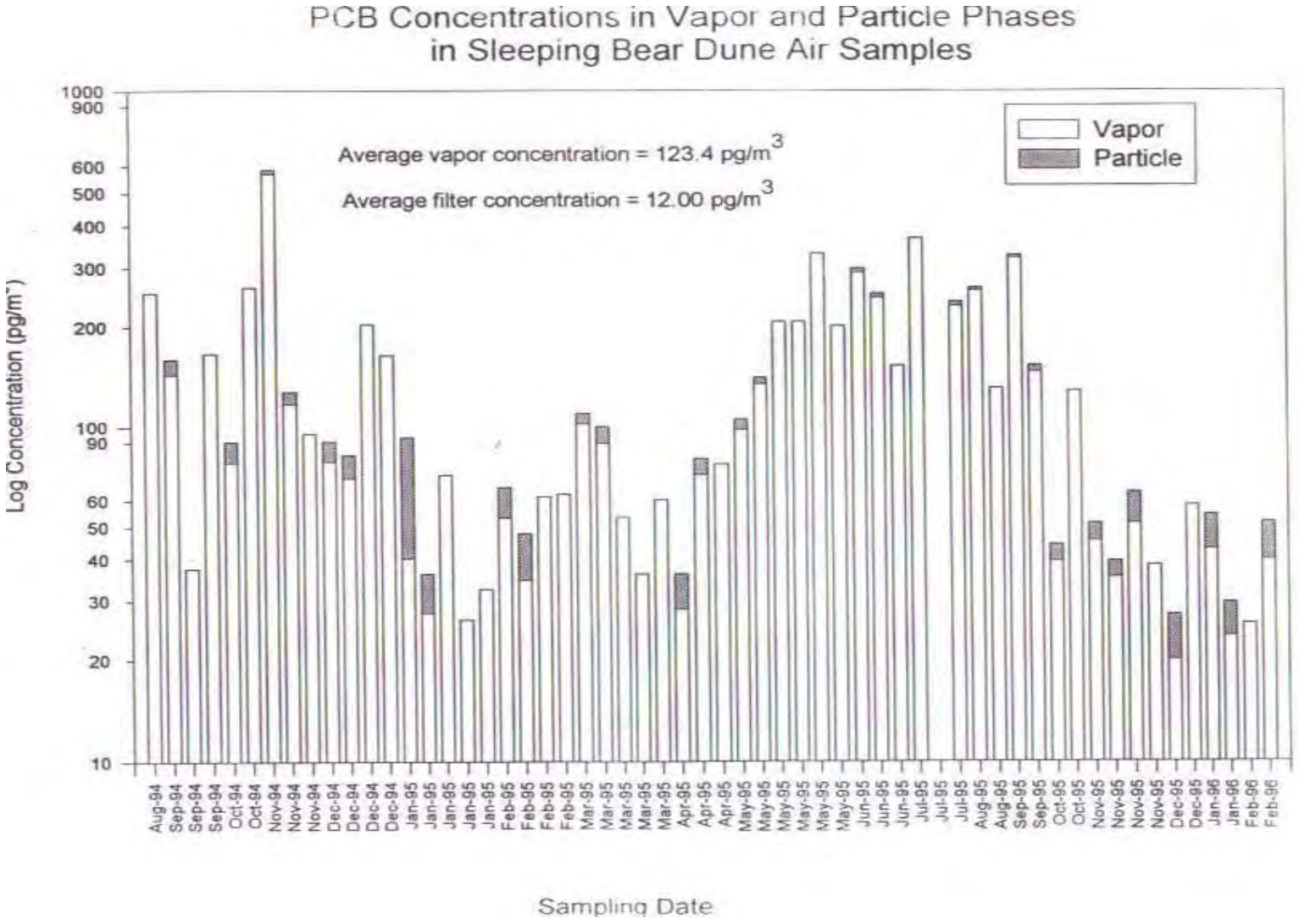
Jan-96
Jan-96
Feb-96
Feb-96


PCB Concentrations in Vapor and Particle Phases
in Sleeping Bear Dune Air Samples
Average vapor concentration = 123.4 pg/m3
Average filter concentration = 12.00 pg/m3
Vapor
Particle
Log Concentration (pg/m3)
1000
900
600
500
400
300
200
100
90
60
50
40
30
20
10
Aug-94
Sep-94
Sep-94
Sep-94
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Oct-95
Oct-95
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Nov-95
Dec-95
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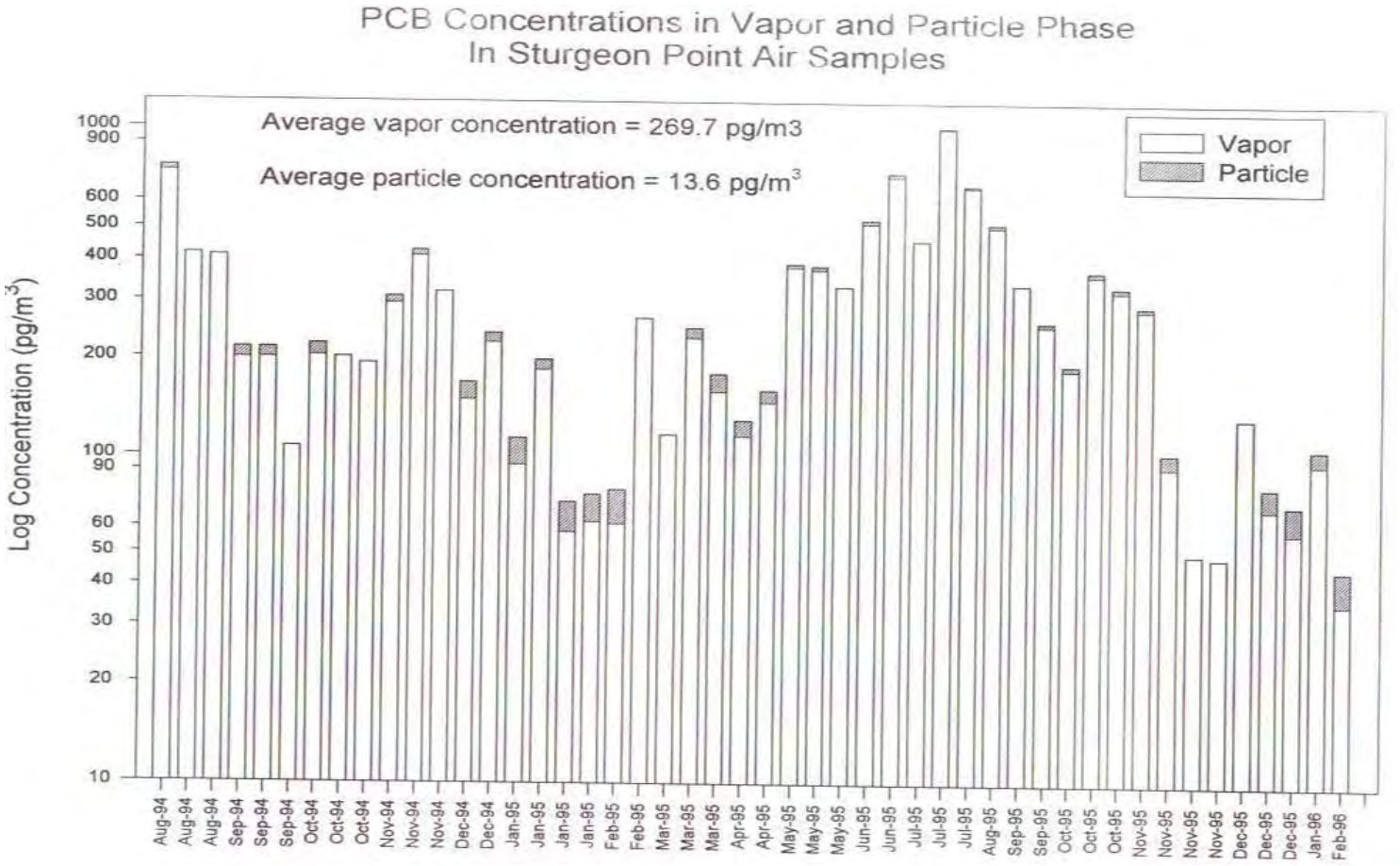
Jan-96
Jan-96
Feb-96
Feb-96
Sampling Date


PCB Concentrations in Vapor and Particle Phase
In Sturgeon Point Air Samples
Average vapor concentration = 269.7 pg/m3
Average particle concentration = 13.6 pg/m3
Vapor
Particle
Log Concentration (pg/m3)
1000
900
600
500
400
300
200
100
90
60
50
40
30
20
10
Aug-94
Aug-94
Aug-94
Sep-94
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Jan-96
Feb-96

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
GREAT LAKES NATIONAL PROGRAM OFFICE
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

DEC 0 3 1996

Dr. Ronald Hites
School of Public Affairs and
Department of Chemistry
Indiana University
Bloomington, IN 47405

Subject: Request to modify the Indiana University IADN Quality Assurance Project Plan

Dear Ron:

This letter is in response to your letter dated November 13, 1996, in which you request the following changes to the project: 1. terminating the compositing of filters for PCB analysis; and 2. terminating the analysis of PAHs with molecular weights of less than 200.

Based on discussions with my IADN counterparts in Canada, I believe that we can justify not compositing the filters to obtain PCB concentrations on particulate matter. The data show that these levels are extremely low, even in the winter. Please document the technical, logistical and financial reasons this will be done in your update of the QA Plan.

At this time, I request that you do not stop analyzing PAHs under molecular weights of 200. I will be considering that option in discussions with the IADN Steering Committee in the future.

If you have any questions, please call me at 312-886-6858.

Sincerely,

A. V. Bandemehr

Angela Bandemehr
IADN Project Officer

cc: Louis Blume

## 17.6. Proposal and Approval of stopping analysis of PCB, HCB, and DDE on filters

SCHOOL OF PUBLIC
AND ENVIRONMENTAL
AFFAIRS

Subject: Revised request for pesticide analysis on filter samples

Dear Angela,

As you know, we have recently stopped compositing the filters and started
tracting and analyzing the individual filter samples. At the same time, we
stopped analyzing PCBs on the filters. In a letter dated March 4, 1997, I a
you to approve discontinuing the analysis of *all pesticides* on the filter sam
because about 90% of the total pesticides (except for dieldrin) are found in
vapor phase.

After further calculations of the dry deposition loadings and of the vapor p
absorption of the pesticides in water (as you suggested), we have decided t
tinue the analyses for most of the pesticides; these are α-HCH, γ-HCH, α-
chlordane, γ-chlordane, *t*-nonachlor, dieldrin, DDT, and DDD.

Of these pesticides, dieldrin is the only one which is detected in all filter s
from all sites. Moreover, about 40% of the total dieldrin is deposited to th
Great Lakes by the particle phase. Therefore, we have decided to continue
eldrin analysis. Dry deposition for the other pesticides, especially the HC
minimal (see the Table), but all of the aforementioned pesticides elute in th
ond (50%) fraction together with dieldrin. Thus, when this fraction is inje
the GC for the analysis of dieldrin, the other pesticides can be measured w
only some additional computer work. I remind you that the Canadians do
analyze any pesticides on the particle filters.

In this letter, we are requesting that we stop analyzing two pesticides in th
extracts. These two compounds are hexachlorobenzene (HCB) and DDE.
offer the following justification:

1. HCB and DDE are rarely detected above the MDL in the filter samples
During the period August 1994-May 1996, out of the 109 samples at E
Harbor, Sleeping Bear Dune, and Sturgeon Point, only 3% had concen
of HCB or DDE above the MDL. (These were all at the most urban si
Sturgeon Point, and for DDE only.)

Bloomington, Indiana
47405-2100

Fax: 812-855-7802

2. Only 0.1% of the HCB and 4-10% of the DDE are due to the particle phase; the rest are due to the vapor phase. Moreover, DDE measurements on the filter samples are not very reliable because 97% are below the MDL.
3. Relative contributions of the gas phase and the particle phase to the loading of these two pesticides to the Great Lakes, show that only 1% of the total HCB is being deposited by particle phase. For DDE, it varies from 4.5 to 35%. This higher percent of deposition and wide range of variability is caused by the very low concentrations, which are almost at the detection levels.
4. Both the pesticides elute in the hexane fraction with PCBs. Since we have stopped analyzing the PCBs on filters, we are not injecting this fraction. Analyses of HCB and DDE will require a whole set of additional instrumental injections and considerable amount of computer analysis just to conclude that these two compounds are not detected.

Therefore, if you please approve this revised proposal to stop analyzing these two pesticides (HCB and DDE), we can stop the collection and analysis of the hexane fraction, which will save some money and time without loosing any meaningful data.

If you have questions please call me at 812-855-2926.

Sincerely yours

Ilora Basu

Ilora Basu
Research Scientist, IADN Project

IB/rah/hs

cc: Dr. Ronald A. Hites
Distinguished Professor

## Dry Deposition versus Absorption to the Great Lakes (kg/yr)

| Pesticides | Eagle Harbor | | | Sleeping Bear | | | Sturgeon Point | | | Average % Dry |
|---|---|---|---|---|---|---|---|---|---|---|
| | Dry | Absorption | % Dry | Dry | Absorption | % Dry | Dry | Absorption | % Dry | |
| α-HCH | 10 | 309 | 3.1 | 7 | 180 | 3.7 | 2 | 93 | 2.1 | 2.8 |
| γ-HCH | 3 | 45 | 6.3 | 2 | 66 | 2.9 | 1 | 20 | 4.8 | 4.7 |
| HCB | 0.4 | 56 | 0.7 | 0.3 | 33 | 0.9 | 0.2 | 15 | 1.3 | 1.0 |
| Dieldrin | 19 | 23 | 45 | 22 | 33 | 40 | 10 | 15 | 40 | 42 |
| p,p'-DDT | 0.7 | 6 | 10 | 1 | 8 | 11 | 2 | 5 | 28 | 16 |
| p,p'-DDD | 0.1 | 2 | 4.8 | 1 | 2 | 33 | 1 | 2 | 33 | 24 |
| p,p'-DDE | 1.1 | 2 | 35 | 2 | 4 | 33 | 1 | 2 | 4.5 | 24 |

These are the approximate relative contributions of the gas and particle phases to the loading to the Great Lakes. No attempt has been made to incorporate accurate meteorological data into the spreadsheet for these calculations. Loadings are based on average pesticide concentrations for the period August 1994 to May 1996. These numbers reflect the relative importance of each phase to the loading to the Great Lakes. Subsequent volatilization has not been considered here.

Date: Fri, 11 Apr 1997 14:19:30 -0400
From: ANGELA BANDEMEHR <BANDEMEHR.ANGELA@EPAMAIL.EPA.GOV
To: hitesr@indiana.edu, ilora@indiana.edu
Subject: filters

Ron and Ilora,

Your fax of April 9 was informative and I agree with you
think we should stop analyzing for HCB and DDE in filter
thus stop the need to collect the hexane fraction. If y
update the necessary QA documents with the appropriate j
(i.e. what you sent me) so that we can keep track of our
process.

Thank you,
Angela

## 17.7. Proposal and Approval of reducing numbers of QC samples

Ms. Angela Bandemehr
United States Environmental Protection Agency
Great Lakes National Program office
77 W. Jackson Boulevard
Chicago, Illinois 60604

SCHOOL OF PUBLIC AND ENVIRONMENTAL AFFAIRS

Subject: Request to modify the Quality Assurance Project Plan

Dear Angela

In response to Ron's e-mail to you, dated May 31, 1996, I am sending this letter requesting that you modify our Quality Control Assurance Plan (QAPP), Revision 3, which was submitted on 7/18/95 and approved on 7/26/95 by Jackie Bode and Michael Papp. By looking at one year's data in our laboratory and from a research point of view we are proposing a small reduction in number of QC samples. These requests are as follows:

1. Setting up one laboratory blank per two batches of samples ( 20- 24 samples) instead of one in each batch of samples (10-12 samples).

2. Setting up one matrix spike per 2 batches of samples (20-24 samples) instead of one in each batch of samples (10-12 samples).

3. Cutting down the collection of field duplicates from 20% to 10%.

4. Cutting down the laboratory duplicates from 10% to 5%.

This will save some time which can be utilized on the loading calculations. Please call me if you have questions at 812-855-2926.

Sincerely yours

Ilora Basu

(Ilora Basu)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
GREAT LAKES NATIONAL PROGRAM OFFICE
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

JUL 23 1996

Ilora Basu
School of Public Affairs and
Department of Chemistry
Indiana University
Bloomington, IN 47405

Subject: TSP/TOC in IADN project

Dear Ilora:

This letter is in response to your letter dated June 27, 1996, in which you request the following changes to the project: 1. decreasing the number of QA/QC samples; and 2. eliminating the TSP/TOC measurments.

First, I have final approval from Lou Blume, our QA officer, that the number of QA/QC samples can be decreased. Please document the technical, logistical and financial reasons this was done in your update of the QA Plan.

Secondly, I am approving the elimination of TSP/TOC measurements. Based on the IADN Steering Committee meeting in Toronto, Canada, earlier this year, there is no reason to continue doing these analyses. Again, please document the technical, logistical and financial reasons for this change in the QA Plan and Project Plan.

If you have any questions, in my absence, please call Lou Blume (312-353-2317).

Sincerely,

A. T. Bandemehr

Angela Bandemehr
Environmental Engineer

## 17.8. Proposal and Approval of reduction of field trip for sampler calibration

INDIANA UNIVERSITY

SCHOOL OF PUBLIC AND ENVIRONMENTAL AFFAIRS

November 1, 1998

Ms. Angela Bandemehr
United States Environmental Protection Agency
Great Lakes National Program office
77 W, Jackson Boulevard
Chicago, Illinois 60604

Subject: Field Equipment Calibration

Dear Angela

This is a request for reducing the frequency of calibration of field equipment at the Integrated Atmospheric Deposition Network (IADN) sites.

As you are aware, Mr. Matt O'Dell makes several trips to the IADN master and satellite stations to calibrate the equipment and to do other routine maintenance. We are operating five sites, and he goes to each site every season. Altogether he makes about 20 trips per year for regular maintenance. Sometimes he has to make additional trips for emergency repair work. We were wondering if we could reduce the number of trips.

We have attached "Quarterly Site Visit Duties" describing what Matt does at the site at each season. The fall and spring visits are most critical when he adjusts the equipment for winter and when he recovers the equipment from winter. Summer is the only time when he replaces the instruments in the Meteorological Tower. For these three seasons, he spends about 1-1.5 days at each site per visit. In winter, he does not have much to do, and spends only 0.5 days at each site.

We have also attached Table 1 showing the percent differences of flow rates in each sampler from season to season.

Bloomington, Indiana
47405-2100

Fax: 812-855-7802

C:\Documents\Letter\ANGELA13.doc

During each visit, Matt measures the flow rates of each sampler and adjusts them to 34 $m^3$ per hour. During the next visit, he measures the flow rates of the same samplers again before doing any adjustment, and he calculates the deviation from th previous setting. Thus, the percent differences of flow rates over a period of 3 months for all samplers are estimated. Table 1 shows these data for 2 years starting from April 1995 through July 1997. According to the IADN Quality Control Quality Assurance Project Plan, the acceptance limit is 10% difference. About 97% of our data are within acceptance limits.

Since Matt's list of duties is shortest in winter, we are wondering if these trips can be omitted unless he gets an emergency call from the site operator. The other option is to do the calibration once in four months instead of three months.

Thanks,

Truly Yours

Ilora Basu

Ilora Basu
Research Scientist
Tel: 812-855-2926
Ilora@Indiana.edu

C:\Documents\Letter\ANGELA

Table 1
Percent Difference (PD) of Sampler Flow Rate

| Eagle Harbor | PD 4/95 | PD 7/95 | PD 10/95 | PD 1/96 | PD 4/96 | PD 7/96 | PD 10/96 | PD 1/97 | PD 4/97 | PD 7/97 |
|---|---|---|---|---|---|---|---|---|---|---|
| TSP | **-2.5** | **0.0** | **-5.0** | **-2.5** | **-5.0** | **-3.8** | **a** | **a** | **a** | **a** |
| Organics Hi Volume #1 | **-2.5** | **-2.5** | **-10.0** | **-5.0** | **-2.5** | **2.5** | **-7.5** | **-10.0** | **-5.0** | **0.0** |
| Organics Hi Volume #2 | **15.0*** | **0.0** | **-2.5** | **5.0** | **-5.0** | **0.0** | **-2.5** | **-7.5** | **-5.0** | **-7.5** |

| Sleeping Bear | PD 4/95 | PD 7/95 | PD 10/95 | PD 1/96 | PD 4/96 | PD 7/96 | PD 10/96 | PD 1/97 | PD 4/97 | PD 7/97 |
|---|---|---|---|---|---|---|---|---|---|---|
| TSP | **-7.5** | **0.0** | **2.5** | **2.5** | **2.5** | **1.3** | **a** | **a** | **a** | **a** |
| Organics Hi Volume #1 | **0.0** | **10.0** | **10.0** | **5.0** | **-10.0** | **7.5** | **+7.5** | **0.0** | **-10.0** | **-2.5** |
| Organics Hi Volume #2 | **-15.0*** | **-5.0** | **-2.5** | **-2.5** | **-2.5** | **-10.0** | **-5.0** | **-2.5** | **-10.0** | **-7.5** |

| Sturgeon Point | PD 4/95 | PD 7/95 | PD 10/95 | PD 1/96 | PD 4/96 | PD 7/96 | PD 10/96 | PD 1/97 | PD 4/97 | PD 7/97 |
|---|---|---|---|---|---|---|---|---|---|---|
| TSP | **3.8** | **2.5** | **5.0** | **2.5** | **3.8** | **2.5** | **a** | **a** | **a** | **a** |
| Organics Hi Volume #1 | **-7.5** | **-7.5** | **-7.5** | **-7.5** | **-7.5** | **-10.0** | **-7.5** | **-5.0** | **-5.0** | **-10.0** |
| Organics Hi Volume #2 | **-5.0** | **5.0** | **5.0** | **10.0** | **-5.0** | **-5.0** | **0.0** | **-2.5** | **-15.0*** | **0.0** |

- **Outside acceptable limits.**
- **[a] The collection of TSP samples was suspended after August 1996.**

Table 1 (continued)

Percent Difference (PD) of Sampler Flow Rate

| IIT Chicago | PD 12/95 | PD 4/96 | PD 7/96 | PD 10/96 | PD 1/97 | PD 4/97 | PD 7/97 |
|---|---|---|---|---|---|---|---|
| TSP | **0.0** | **1.3** | **1.3** | [a] | [a] | [a] | [a] |
| Organics Hi Volume #2 | **-5.0** | **-5.0** | **-7.5** | **0.0** | **10.0** | **10.0** | **-5.0** |

| Brule River | PD 1/96 | PD 4/96 | PD 7/96 | PD 10/96 | PD 1/97 | PD 4/97 | PD 7/97 |
|---|---|---|---|---|---|---|---|
| TSP | **-1.3** | **-2.5** | **-1.3** | [a] | [a] | [a] | [a] |
| Organics Hi Volume #1 | **-5.0** | **2.5** | **2.5** | **0.0** | **-10.0** | **0.0** | **-10.0** |

[a] **The collection of TSP samples was suspended after August 1996**

## Quarterly Site Visit Duties

| Summer Visit | | Winter Visit | |
|---|---|---|---|
| Rain Gage | 1-2 hrs | Rain Gage | 0 hrs |
| Calibrate Rain Gage | | Rain Gage is not calibrated in winter | |
| Organics Hi-Vol Sampler* | 1 hr | Organics Hi-Vol Sampler* | ½ - 1 hr |
| Check flow rate of motor<br>Replace motor if necessary<br>Calibrate/Set flow with new motor | | Check flow rate of motor<br>Calibrate/Set flow of motor | |
| Dichot | 1 hr | Dichot | 1 hr |
| Calibrate Dichot | | Calibrate Dichot | |
| MIC* | ½-1 hr | MIC* | ½-1 hr |
| Check operation<br>Check/replace seating pad<br>Adjust heater and fan | | Check operation<br>Check/replace seating pad<br>Adjust heater and fan | |
| Met Tower | 2-3 hrs | Met Tower | 0 hrs |
| Replace Relative Humidity, Wind Speed and Wind Direction instruments. | | Met Tower work is done in the summer. | |

Miscellaneous repairs, cleaning and replenishing supplies 0-1 hr

**Total for Summer** **5½ - 9 hours (Satellite Sites)**
**7 – 10½ hours (Master Stations)**

**Total for Winter** **2 – 3 hours (Satellite Site)**
**3 – 4 hours (Master Station)**

## Quarterly Site Visit Duties

| Spring Visit | | Fall Visit | |
|---|---|---|---|
| Rain Gage | 2-3 hrs | Rain Gage | 2-3 hrs |

Date: Wed, 16 Dec 1998 10:44:43 -0600
From: ANGELA BANDEMEHR <BANDEMEHR.ANGELA@epamail.epa.gov>
To: ilora@indiana.edu
Cc: hitesr@indiana.edu, jaodell@indiana.edu
Subject: Chicago site and Matt's trip -Reply

Illora,
I will call Nasreen, who left a message for me yesterday. She says she cannot reach you at the phone number you left. You might want to contact her again.
As for Matt's trips, I haven't heard much either, but if Ray has no problem with it, neither do I. Go ahead and plan to make the change. I hearby authorize it.

Angela

>>> ilora basu <ilora@indiana.edu> 12/15/98 03:10pm >>>

Dear Angela

I have talked to Dr. Tom Holsen this morning about Nasreen Khalili and he thinks that she is a very suitable person for IADN air sampling project at Chicago. After that, I have talked to Nasreen who is a faculty member at IIT and
talked about our project. Her student, Hitendra Jain, is going to replace Nedim from next month. Nasreen seems to be very willing to participate in this project. I got all information about Hitendra and start paper work soon. Nedim already has started training Jain from this morning.

Have you taken any decision about Matt's field trip? We got one response from Ray Hoff that he is willing to accept this change. Please let me know your final decision. We will plan accordingly.

Best wishes

Ilora Basu
SPEA 410
Indiana University
Bloomington
Indiana 47405

## 17.9. Proposal and Approval of Omitting MIC wipes

INDIANA UNIVERSITY

April 19, 2000

Ms. Melissa Hulting
United States Environmental Protection Agency
Great Lakes National Program office
77 W, Jackson Boulevard
Chicago, Illinois 60604

SCHOOL OF PUBLIC AND ENVIRONMENTAL AFFAIRS

Subject: Change in procedure for precipitation sample extraction.

Dear Melissa

We propose to make a minor change in our precipitation sample extraction procedure. All these years we have combined the wet filter papers that have been used to wipe off the MIC sampler funnels with the XAD-2 rain columns. Thus, we extracted and analyzed the wipe and XAD column together. The wipes are supposed to catch the last part of the particles that were sticking to the funnels after the 28 day collection period. Following this procedure, we did not have any problem with recoveries of compounds of interest from the matrix spike experiments or recoveries of surrogate standards from each sample. Table 1 shows the recoveries of PCB surrogates (congener 14, 65, and 166) and Graph 1 shows the recoveries of pesticide and PAH surrogates ( dibutylchlorendate and $d_{10}$ phenanthrene).

Recently, we added δ-HCH as a new surrogate standard for the pesticides and found that water in the wet filter paper wipes is interfering with the recoveries of this compound. It is very likely that the same water has interfered with the recoveries of other HCHs. For example, in 1998, the results from the

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47405-2100

interlaboratory comparison study at Point Petre showed that EHD and NWRI in Canada detected more α-HCH and γ-HCH in precipitation samples than IU. The results are plotted in Graph 2.

NWRI has stopped combining the wipes with XAD-2, because they could not detect any compound of interest on the wipes. We have analyzed the wipes separately and found only blank level of PCBs (0.3 to 6 ng) and practically no pesticides and PAHs. All the compounds are in the XAD-2 column. By extracting the XAD-2 column only, our δ-HCH recoveries improved from 8% to 85%. Results are plotted in Graph 3. Our values of α-HCH and γ-HCH in the samples also improved. Thus, by adding wet wipes in our samples, we are reducing the extraction efficiency of HCHs and at the same time adding some extra PCBs to our samples. We thought about the possibility of dry wipes, which worked well for recoveries, but depending on the rain event, it is not always possible for the site operators to keep the wipes dry.

Therefore, in our opinion it will be best to omit the wipes completely. The Canadians already do this. We have started separating the XAD-2 and the wipes and storing the wipes in the cold room. After we get your approval, we will switch to analyzing the XAD-2 only and get rid of all the wipes.

Thank you

Ilora Basu

Ilora Basu
Research Scientist
SPEA 410
Tel: 812-855-2926
E-mail: Ilora@Indiana.edu

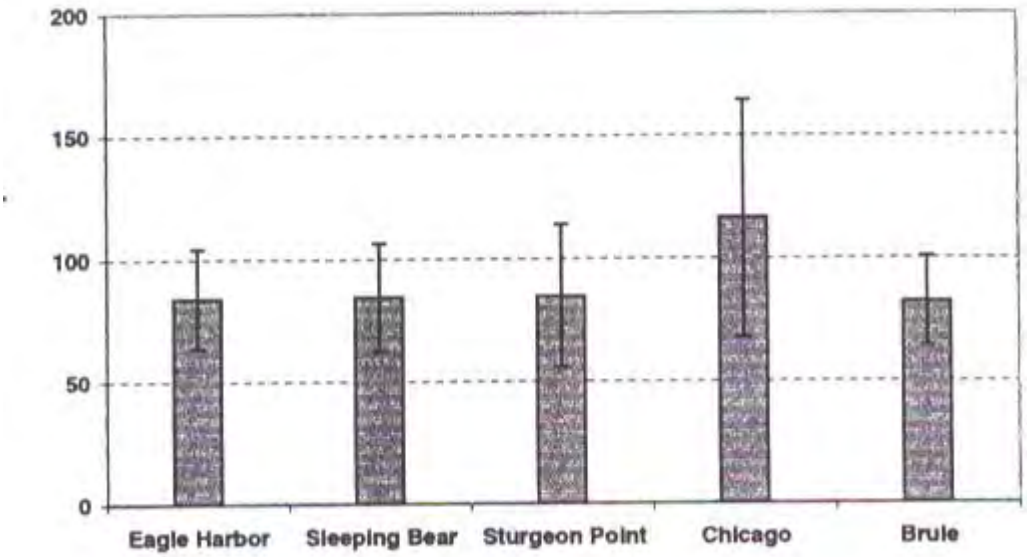
**Table 1**

**PCB Surrogate Percent Recoveries from Precipitation Samples
1994-1998**

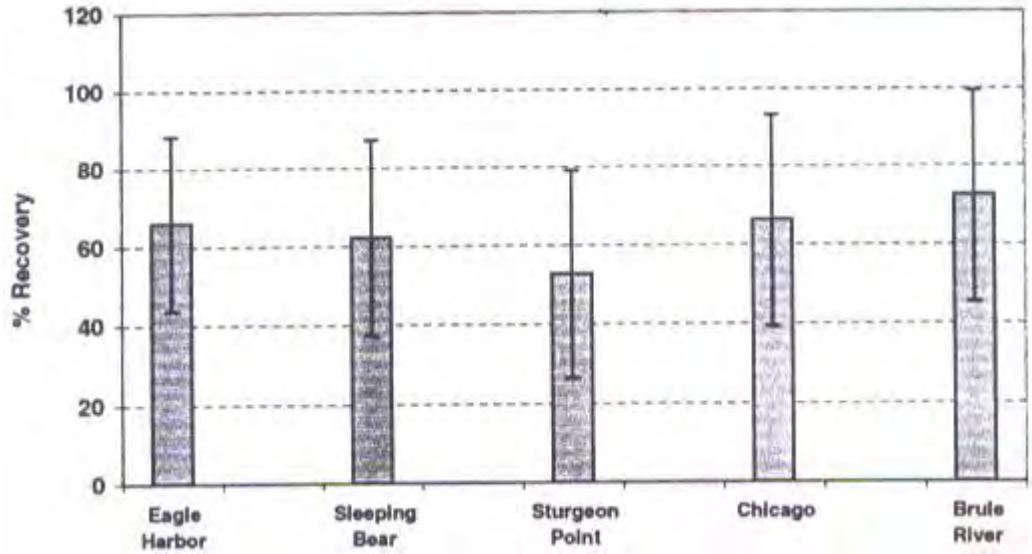
| Surrogates | Eagle Harbor | Sleeping Bear Dunes | Sturgeon Point | Brule River | Chicago |
|---|---|---|---|---|---|
| Congener 14 | 93.99 | 95.85 | 93.4 | 96.88 | 99.6 |
| Congener 65 | 89.71 | 90.34 | 87.09 | 89.7 | 90.02 |
| Congener 166 | 95.32 | 96.68 | 94.3 | 96.47 | 100.09 |

Graph 1

Pesticide Surrogate (DBC) Percent Recovery from Precipitation Samples
1994-1998

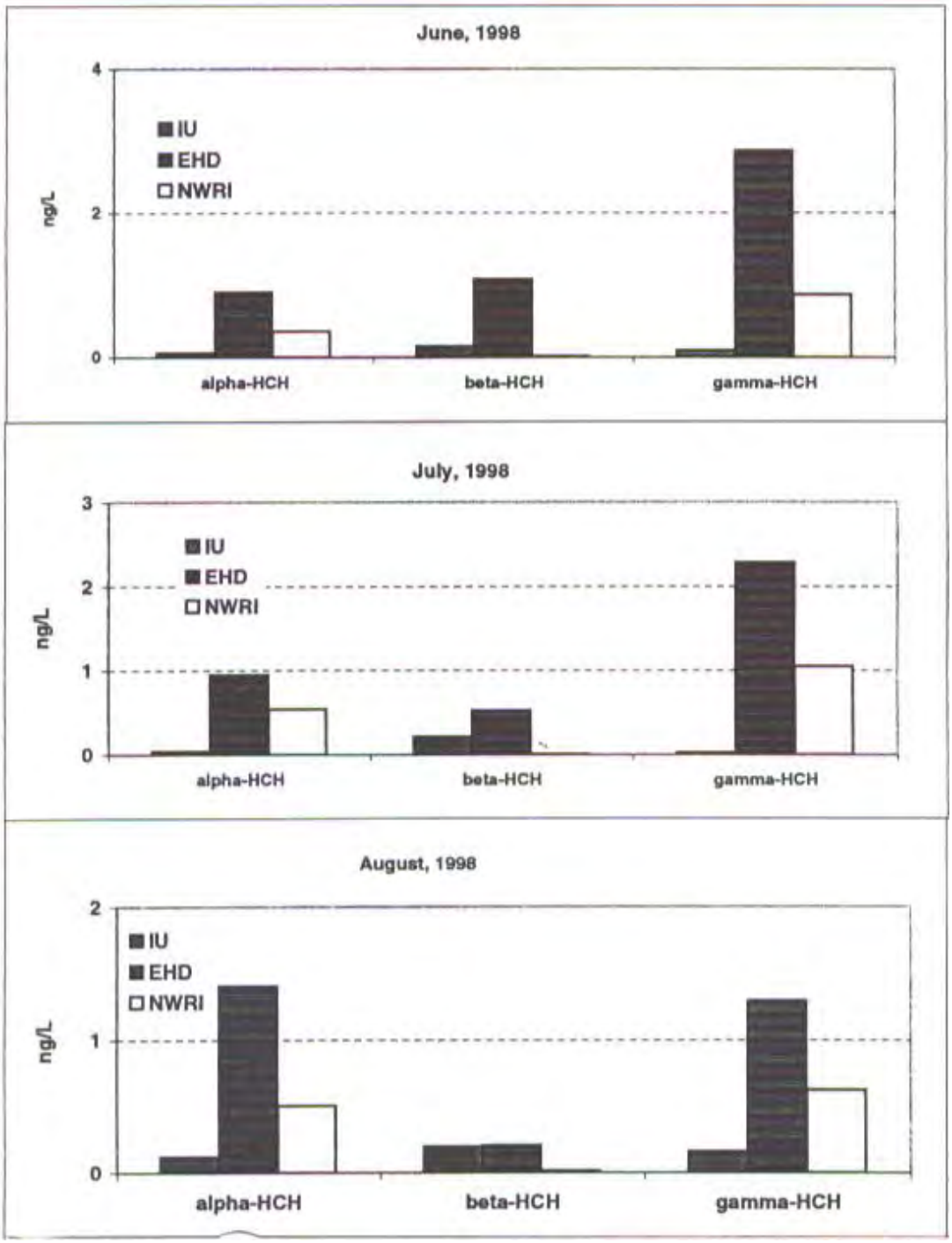


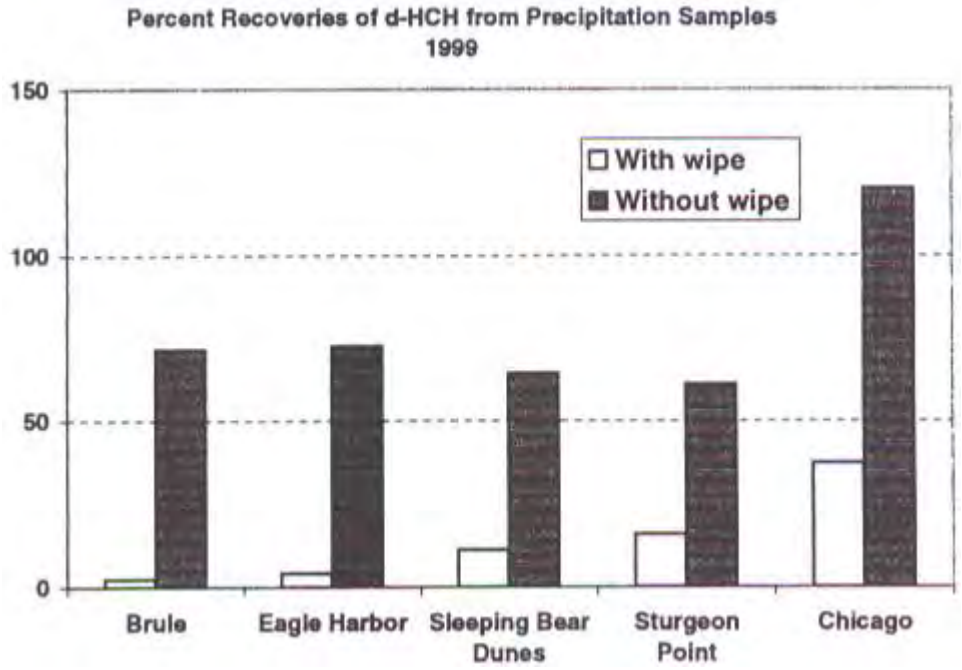

PAH Surrogate ($d_{10}$ Phenanthrene) Percent Recoveries from Precipitation Samples
1994-1998



**Graph 2**

**Interlaboratory comparison of HCHs in Point Petre Precipitation Samples**




Percent Recoveries of d-HCH from Precipitation Samples
1999
150
100
50
0
With wipe
Without wipe
Brule
Eagle Harbor
Sleeping Bear Dunes
Sturgeon Point
Chicago

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
GREAT LAKES NATIONAL PROGRAM OFFICE
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

Ilora Basu
School of Public and Environmental Affairs
SPEA 410
1315 E. 10th Street
Indiana University
Bloomington, Indiana 47405

Subject: Change in procedure for precipitation sample extraction

Dear Ilora:

This is in response to your letter of April 19, 2000 in which you request to quit analyzing the wet filter wipes used to wipe the MIC sampler funnel. I understand that the wipes have been separated from the XAD and stored since June 1999. I am approving the change, effective for samples from that date on. Therefore, the stored wipes may be discarded. Please document the logistical, technical, and/or financial reasons for this procedure change in future updates of your Quality Assurance Project Plan.

You noted that you had no recovery problems prior to the addition of δ-HCH as a surrogate standard. However, you suspect that the water in the wipes may have interfered with recovery of other HCHs in the past. In addition, data from a November 1999 sample that you sent shows poor recovery of all surrogates when the wipe was analyzed separately. Any significant increases/changes in recoveries for post-June 1999 samples should be noted and given explanation in future data and results reporting in order to ensure data comparability over the entire IADN monitoring period.

If you have any questions, you may contact me at (312) 353-3612 or the GLNPO Quality Assurance Officer, Lou Blume, at (312) 353-2317.

Sincerely,

Paul J. Horvatin
Project Officer

Printed on Recycled Paper

## 17.10. Proposal and Approval of Stopping filter pesticide analysis

**From:** Hulting.Melissa@epamail.epa.gov
**Sent:** Monday, January 05, 2004 5:04 PM
**To:** Basu, Ilora
**Cc:** Hites, Ronald A.
**Subject:** Re: FW: filter pesticides

fb vs
ple_melissa.doc (95 k b)

This is old stuff, but did I ever send a message OK'ing dropping the particulate pesticides?

"Basu, Ilora" <ilora@indiana.edu> — To: Melissa Hulting/R5/USEPA/US@EPA; cc: "Hites, Ronald A." <hitesr@indiana.edu>; Subject: FW: filter pesticides; 05/19/2003 01:46 PM

Dear Melissa,

Ron told me that you did not get my e-mail message on filter pesticides. I am resending my previous message. I am also sending you 2 tables from 2001 QC report. One is sample to blank ratio of filter pesticides. About 90% of them are lower than 5 times. I have highlighted some numbers where the ratios are higher than 5. They seem to be lower in 2002 samples. The 2nd table is on sample to blank ratio of vapor pesticide. I added this one so that you can see the differences between the two matrices.

I think we can stop analyzing the filters for pesticides except for the urban area like Chicago and Cleveland.

Best wishes,

Ilora

-----Original Message-----
From: Basu, Ilora
Sent: Monday, April 07, 2003 4:34 PM
To: 'Hulting.Melissa@epamail.epa.gov'
Cc: Hites, Ronald A.
Subject: RE: notes from March 12 IADN call-reply

Dear Melissa,

I read the minutes from March 12th call. I have some thoughts about getting rid of the OC pesticides. It is true, that if we get rid of some pesticides, it is not going to help much. But it depends on how many you are getting rid off. If you get rid of just 2 it is not going to help. But if you can get rid of 5 or 6 it will help. Because, we hand correct every peak area. Some pesticides like o,p'DDT, aldrin, endrin are not seen much except for the Chicago samples. Again, these are often

interfered with some other peaks in DB5. So if we get high values for these compounds we need to run these samples again in 1701 column for confirmation. Finally, we omit the data because DB5 results and 1701 results do not agree. These are no doubt problem pesticides for us. Can we stop the confirmation work on these pesticides?

The other concern is the particle pesticides. We are getting very low values. So, it is time to think about stopping the particle pesticide work. We still can continue analyzing the Chicago and Cleveland filters.

I have discussed this matter with Ron. You may want to raise this issue at Egbert meeting in May.

Best wishes,

Ilora

Ilora Basu
School of Public and Environmental Affairs
SPEA 471
1315 E 10th Street
Indiana University
Bloomington, IN 47405

Phone: 812-855-2926
Fax: 812-855-1076

-----Original Message-----
From: Hulting.Melissa@epamail.epa.gov [mailto:Hulting.Melissa@epamail.epa.gov]
Sent: Monday, March 31, 2003 3:51 PM
To: Pierrette.Blanchard@ec.gc.ca; c.h.chan@ec.gc.ca; Frank.Froude@ec.gc.ca; Basu, Ilora; Ken.Brice@ec.gc.ca; Melanie.Neilson@ec.gc.ca; Hites, Ronald A.; Celine.Audette@ec.gc.ca; nettesheim.todd@epamail.epa.gov; pfowlie@cogeco.ca; Ed.Sverko@cciw.ca; Cathy.Banic@ec.gc.ca
Subject: notes from March 12 IADN call

Attached are the minutes from our last call.

Please forward any more comments on the peer review report to the group. After I receive the final report from the panel, I am going to start drafting a response, so your input is very important.

Melissa

(See attached file: 031203PeerReviewResp.doc)
(See attached file: fb vs sample_melissa.doc)

Table 4.16

Sample/ Blank Concentration

Particle Pesticides

| | Eagle Harbor | Sleeping Bear Dunes | Sturgeon Point |
|---|---|---|---|
| | Sample:Blank | Sample:Blank | Sample:Blank |
| alpha-HCH | 0.82 | 0.37 | 1.70 |
| beta-HCH | 3.34 | #DIV/0! | 0.10 |
| gamma-HCH | 1.11 | 1.42 | 0.87 |
| heptachloroepoxide | 2.29 | 1.20 | 1.34 |
| oxychlordane | 6.20 | 1.66 | 3.46 |
| gamma-chlordane | 1.50 | 1.69 | 1.64 |
| endosulfan I | 5.13 | 2.55 | 2.97 |
| alpha-chlordane | 0.88 | 2.00 | 8.01 |
| trans-nonachlor | 2.11 | 1.15 | 2.94 |
| dieldrin | 4.20 | 1.92 | 3.40 |
| o,p-'DDD | 0.41 | 0.94 | 1.81 |
| endrin | 1.81 | 1.09 | 4.85 |
| endosulfan II | 13.75 | 4.62 | 7.54 |
| p,p'-DDD | #DIV/0! | 0.69 | 4.77 |
| endosulfan sulfate | 17.43 | 6.74 | 7.15 |
| p,p'-DDT | 2.21 | 2.54 | 2.16 |
| methoxychlor | #DIV/0! | 2.35 | #DIV/0! |

## Table 4.14

## Sample: Blank Concentrations

## Vapor pesticides

| | Eagle Harbor | Sleeping Bear Dunes | Sturgeon Point |
|---|---|---|---|
| | Sample:Blank | Sample:Blank | Sample:Blank |
| alpha-HCH | 490.11 | 205.90 | 776.09 |
| beta-HCH | 21.65 | 17.23 | 72.66 |
| gamma-HCH | 201.16 | 174.09 | 477.45 |
| heptachloroepoxide | 50.38 | 63.93 | 122.04 |
| oxychlordane | #DIV/0! | #DIV/0! | #DIV/0! |
| gamma-chlordane | 51.95 | 21.17 | 91.08 |
| endosulfan I | 120.80 | 140.71 | 242.71 |
| alpha-chlordane | 10.87 | 33.29 | 30.41 |
| trans-nonachlor | 49.48 | 55.63 | 198.12 |
| dieldrin | 110.67 | 82.16 | 172.74 |
| o,p-'DDD | 13.30 | 18.50 | 12.41 |
| Endrin | 5.39 | 10.33 | 4.33 |
| endosulfan II | 430.87 | #DIV/0! | #DIV/0! |
| p,p'-DDD | 2.98 | #DIV/0! | #DIV/0! |
| endosulfan sulfate | #DIV/0! | 3.63 | 10.87 |
| p,p'-DDT | 13.43 | 38.95 | 375.74 |
| methoxychlor | #DIV/0! | 19.74 | #DIV/0! |
| HCB | 247.19 | 211.06 | 367.69 |
| p,p'-DDE | 180.98 | 468.88 | 461.08 |
| Aldrin | #DIV/0! | #DIV/0! | #DIV/0! |
| o,p'-DDT | #DIV/0! | #DIV/0! | #DIV/0! |
| octachlorostyrene | #DIV/0! | #DIV/0! | #DIV/0! |

## 17.11. Proposal and Approval of Stopping Belfort Rain Gauge

**Basu, Ilora**

**From:** Hulting.Melissa@epamail.epa.gov
**Sent:** Wednesday, August 04, 2004 2:03 PM
**To:** Basu, Ilora
**Subject:** RE: FW: Belfort Rain Gauges

You may stop the rain sample collection in the Belfort gauge, as well as the calibration.

"Basu, Ilora" <ilora@indiana.edu>
08/04/2004 11:47 AM

To
Melissa Hulting/R5/USEPA/US@EPA
cc
bcc
Fax to

Subject
RE: FW: Belfort Rain Gauges

Melissa,

Thank you very much for stopping the Belfort calibration. Just for record keeping's sake, please send me a line stopping the rain sample collection in Belfort. Although stopping calibration means stopping sample collection. But it will be nice to keep the record straight.

Thanks,

Ilora

Ilora Basu
Research Scientist
School of Public and Environmental Affairs SPEA 471
1315 E. 10th Street
Indiana University

Bloomington, IN 47405

Phone: 812-855-2926
Fax: 812-855-1076

From: Hulting.Melissa@epamail.epa.gov
[mailto:Hulting.Melissa@epamail.epa.gov]
Sent: Tuesday, July 20, 2004 10:21 AM
To: Hafner, William D
Cc: Basu, Ilora; Hites, Ronald A.
Subject: Re: FW: Belfort Rain Gauges

I know that we don't use the Belfort data, but I did want to know why there was a difference. And now we aren't even using at-station precip amounts for the loadings either.

So stopping the calibration of the Belfort rain gauges is fine. I know Ilora keeps track of such decisions and includes the e-mails/memos in an appendix to the QAPP. I will keep a copy of this as well.

Melissa

-----"Hafner, William D" <whafner@indiana.edu> wrote: -----

To: Melissa Hulting/R5/USEPA/US@EPA
From: "Hafner, William D" <whafner@indiana.edu>
Date: 07/20/2004 10:01AM
Subject: FW: Belfort Rain Gauges

Melissa,
There is a difference that is fairly seasonal. The MIC collects less rain in the winter. We never actually use the rain gauge data for anything. The only important parameter for precipitation is the volume of water collected by the MIC.
Will

-----Original Message-----
From: Carlson, Daniel
Sent: Tuesday, July 20, 2004 9:36 AM
To: Hafner, William D
Cc: Basu, Ilora
Subject: RE: Belfort Rain Gauges

The largest portion of the difference can be attributed to the expected

difference in collection efficiencies of snow. The Belfort has a wind shield in the winter; the MIC does not. We would expect the MIC to collect about 50% of the precipitation in the form of snow compared to the shielded Belfort.
This is about what we actually see. There are also some differences in the summer, for which we have no explanation, but these are relatively minor.

Quoting "Hafner, William D" :

> Dan/Ilora,
> After talking with Ron, I sent an email to Melissa Hulting suggesting > that we quit work on the rain gauges. Do you have an answer to her > question of what the difference is? I realize we don't know what the > reason is, but suspect that it is due partially to snow effects.
> Will
>
>
>
> -----Original Message-----
> From: Hulting.Melissa@epamail.epa.gov > [mailto:Hulting.Melissa@epamail.epa.gov]
> Sent: Monday, July 19, 2004 5:08 PM
> To: Hafner, William D

>
> Melissa,
>
>
> In our most recent (and approved) proposal for IADN, we mentioned that > we no longer want to use the rain gauges (seems as they don't match up > with the MIC volumes, and we have no use for the Belfort data). Is it > OK if we just stop calibrating the gauges on the site visits and close > them up?
>
>
> Thanks.
>
>
> Will
>

## 17.12. Proposal and Approval of Stopping Performance Standard

**Basu, Ilora**

**From:** Hulting.Melissa@epamail.epa.gov
**Sent:** Thursday, December 01, 2005 3:48 PM
**To:** Basu, Ilora
**Cc:** Hites, Ronald A.
**Subject:** elimination of old PCB check standard

Ilora:

As we discussed on Monday, I approve of stopping the use of the old PCB check standard, since we are now using the interagency common reference standard for PCBs. Using both standards as check standards would be duplicative.

Melissa

^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^^

Melissa Hulting
IADN Program Manager
U.S. EPA Great Lakes National Program Office (GLNPO)
77 W Jackson Blvd, Mail Code G-17J
Chicago, IL 60604
312.886.2265
fax 312.353.2018
hulting.melissa@epa.gov

PCB Performance standard was stopped from 12/1/05

Ilora Basu
12/2/05

**From:** Hulting.Melissa@epamail.epa.gov
**Sent:** Friday, December 02, 2005 10:26 AM
**To:** Basu, Ilora
**Subject:** RE: elimination of old PCB check standard

Yes, I approve of eliminating the old check standards for PAHs and OC pesticides since we now have common ref standards for those chemical groups as well.

Melissa

"Basu, Ilora" <ilora@indiana.edu>

12/02/2005 07:11 AM

To
Melissa Hulting/R5/USEPA/US@EPA
cc

Subject
RE: elimination of old PCB check standard

Pesticides and PAH prf. std were stopp from 12/1/05

Ilora Basu

Melissa,

Thanks for the e-mail. Please send me another e-mail mentioning pesticides and PAHs. We are running Peter's C these two analysis too.

Ilora

Ilora Basu

Research Scientist

School of Public and Environmental Affairs

## 17.13. Stopping Temperature Chart Recorder

**Basu, Ilora**

**From:** Bays, James C
**Sent:** Thursday, January 19, 2006 9:31 AM
**To:** Shital Jiwane; Ryan Justice; Carlton See; Darrel Smith; Tom or Alice van Zoeren
**Cc:** Basu, Ilora; osborne@uiuc.edu
**Subject:** Back-Up temperature recorders

IADN Site Operators:

This message is to inform all site operators of a change in status regarding back-up temperature recorders. It has been decided that these units are on no longer necessary to provide such back-up. One reason for this change is due to the difficultly in reading the paper discs used with the unit. Also, with ready access to such data via NOAA or similar web-based services, using the temperature recorder could be considered wasteful. So please send them back to us (Indiana University) at your convenience.

Thanks for your good job

Jim Bays

## 17.14. Stopping PCB Analysis in Precipitation Samples from Remote Sites

**Basu, Ilora**

**From:** Hulting.Melissa@epamail.epa.gov
**Sent:** Friday, June 23, 2006 3:22 PM
**To:** Basu, Ilora
**Cc:** Hites, Ronald A.
**Subject:** Re:

I e-mailed Paul last week asking for a final decision, but he never replied. I was waiting for an OK from him. However, he was okay with it when Ron was here and we discussed it verbally. I've also discussed it with Pierrette, and she approves.

Therefore, I'm going to go ahead and formally approve discontinuing PCB analyses in precipitation for the Eagle Harbor, Sleeping Bear Dunes, and Sturgeon Point samples.

Thanks Ilora--have a good weekend!

Melissa

"Basu, Ilora" <ilora@indiana.edu>
06/23/2006 07:03 AM

To: Melissa Hulting/R5/USEPA/US@EPA
cc: "Hites, Ronald A." <hitesr@indiana.edu>

Subject

Dear Melissa,
In the IAGLR Steering Committee meeting we talked about stopping the PCB analysis in the precipitation samples of the remote sites (EH, SBD, STP). Is there any final decision on that? We have started extraction of the 2006 samples. If we decide to stop the analysis, this is a good time. We are continuing with the pesticide and the PAH analyses. Until we hear from you we will just save the extracts in the freezer. Please let me know. Thanks,

Ilora

Ilora Basu
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1315 E. 10th Street
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Bloomington, IN 47405

Phone: 812-855-2926 (off)
812-856-4364 (lab)

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## 17.15. Reduction of Field Duplicate and Field Blank

**Basu, Ilora**

**From:** Hulting.Melissa@epamail.epa.gov
**Sent:** Thursday, July 06, 2006 12:55 PM
**To:** Hites, Ronald A.; Basu, Ilora
**Cc:** Pierrette.Blanchard@ec.gc.ca; Horvatin.Paul@epamail.epa.gov
**Subject:** Re: Money, money

**Attachments:** Cuts in IADN budget_Approvals MLH PB.doc

Cuts in IADN budget_Approvals ..

Ron and Ilora:

Attached is your letter, with the decisions made by Pierrette and I. We approve the stopping of PCBs in precip at the remote sites (#1) and reduction of frequency of field blanks and duplicates (#3). The other three options still may be considered in the future after further analysis and consultation.

Thank you for your assistance in this activity. Please use the results of these decisions in formation of your budget and proposal for 2006-2007, which I need as soon as is feasible.

Melissa

(See attached file: Cuts in IADN budget_Approvals MLH PB.doc)

"Hites, Ronald A." <hitesr@indiana.edu>
06/30/2006 01:17 PM

To: Melissa Hulting/R5/USEPA/US@EPA
cc: "Basu, Ilora" <ilora@indiana.edu>, "Hites, Ronald A." <hitesr@indiana.edu>

June 30, 2006

Ms. Melissa Hulting
U.S. Environmental Protection Agency
Great Lakes National Program Office

Dear Melissa,

Here are some of our (mine and Ilora's) thoughts about where we could save money on IADN. These are presented in roughly our suggested order of priority.

1. We could (and should) stop measuring PCBs in precipitation at EH, SBD, and SP because the current measurement are so low that they are not much above blank levels at these three sites. This would save about $8,000. I note that we have already made this decision so here are come savings right off the bat.

**Decision: Approved, starting immediately.**

2. We could eliminate a few of the analytes. Eliminating β-HCH, *o,p'*-DDD, endrin, and methoxychlor from our list would allow us to drop the 1701 confirmatory GC analyses and save about $6,000.

**Decision: Not approved. Dropping the specified analytes is not the concern here; rather it's the dropping of the 1701 confirmation. We have seen some differences between columns in our dealings recently with the sudden drop in a-HCH concentrations, which may be "real" or an artifact of switching columns. We should continue to keep tabs on any column differences.**

3. We could stop all sampling at Point Petre. This would reduce our sample count by 20 air samples and 14 precipitation samples for a reduction in cost of about $25,000. After all of these years, we know that our data at this site are about two times higher than the Canadian data. We believe that this trend will continue into the future, and there is little need to beat a dead horse. Alternately, we could temporarily stop these samples, but re-start them later.

**Decision: Not approved at this time. It may be possible to drop this in the future after Rosa's further analysis of the intercomparison data (we would like a few years of data in the EHD clean-lab period included). Also, I (Melissa) believe that EHD and IU are now both analyzing for PBDEs. We should look at any differences in the Point Petre precip samples.**

4. We could cut back on the number of QC samples (including the field blanks and the field duplicated. Now 10% of our samples are QC samples; cutting this to 5% would save about $12,000.

**Decision: Approved. We approve this action since there is a long record of QC samples for IU and we have a sense of blank issues and precision. Our assumption is that personnel within an experienced laboratory like yours will be able to know from the chromatogram/regular samples if something seems off.**

5. We could stop all sampling at Eagle Harbor, and this would save about $40,000. However, on reflection neither Ilora nor I think this is a real good idea. If we did drop this site, we would have to use Burnt Island data for the loading calculations for Lake Superior, and we all know that some of the Burnt Island data suffer from break through problems (PCBs and HCHs for example). We think that sampling once every 24 days would be better than closing this site, and this strategy would save about $20,000.

**Decision: Not approved at this time, but something we could consider in the near future. We would have to consult with the Lake Superior LaMP first, and given that we need the new budget and proposal very soon there is not much time for that. We have already pulled out of Brule River; there may be some political ramifications of pulling out of Lake Superior completely. There is also some value to having to what amounts to a global background station, as well as one closer to the western end of the basin. Lake Superior's inputs are atmospherically driven and the lake has somewhat peculiar bioaccumulation issues in fish tissue given its extreme oligotrophic state.**

The dollar amounts given above a smaller that I had first estimated. You may remember that I said that our costs were directly proportional to the number of samples. Well, I was wrong. I forgot that we have some fixed costs that we cannot cut – these include costs for my time, for Ilora's (now 75%) time, and to some extent, costs for one of the post-docs.

We would be happy to discuss these items 1 to 5 further and to hear your thoughts on what you think we should cut and when. In any case, I remind you that we need to submit our continuation proposal before August 10, when I leave for Europe for 17 days.

Sincerely yours

Ronald A. Hites

Ronald A. Hites
Distinguished Professor

RAH/hs

## 17.16. Moving MIC-2 from Eagle Harbor to Chicago

**Basu, Ilora**

**From:** Hulting.Melissa@epamail.epa.gov
**Sent:** Wednesday, July 12, 2006 2:58 PM
**To:** Basu, Ilora
**Subject:** RE: EGH MIC 2

Sorry Ilora, I temporarily blanked on the fact that if we're not doing the PCBs in precip, we certainly don't need a dup there!

I approve of bringing MIC #2 from Eagle Harbor to Chicago.

Thanks,
Melissa

"Basu, Ilora" <ilora@indiana.edu>
07/12/2006 06:59 AM
To: Melissa Hulting/R5/USEPA/US@EPA
cc:
Subject: RE: EGH MIC 2

Yes, Melissa. We regularly collect FB and FD from Eagle harbor. But we are stopping the PCB analysis from Eagle Harbor precipitation. We have already established a blank data from Eagle harbor. I assumed that reliable blank data from Chicago is more important than Eagle Harbor values. But if you want o keep the Eagle Harbor MIC 2 undisturbed and bring Jerry's MIC to Chicago after modification, that will be ok with me. But before doing that we need to find out if it really belongs to IADN or not.

The bottom line is we need a MIC in Chicago to collect FB and FD. Now which one to bring? MIC2 from EH or Jerry's MIC?

Best wishes,

Ilora

Ilora Basu

Research Scientist

School of Public and Environmental Affairs

SPEA 471 or 456

1315 E. 10th Street

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## 17.17: Throwing away hard copies of old data

**Basu, Ilora**

**From:** Hulting.Melissa@epamail.epa.gov
**Sent:** Monday, April 24, 2006 2:44 PM
**To:** Basu, Ilora; Hites, Ronald A.
**Subject:** Re: FW: IADN & LMLS

I agree with Ron. If it's just hard copy data, let's dump it.
Everything (or vast majority of it) is in the database.

What is the LMLS?

Melissa

"Basu, Ilora"
<ilora@indiana.edu>
04/24/2006 01:29 PM

To: Melissa Hulting/R5/USEPA/US@EPA
cc:

Subject: FW: IADN & LMLS

Melissa,
Please see the trail. Please let me know your opinion. Thanks,

Ilora

Ilora Basu

Research Scientist

School of Public and Environmental Affairs

SPEA 471 or 456

1315 E. 10th Street

Indiana University

Bloomington, IN 47405

Phone: 812-855-2926 (off)

812-856-4364 (lab)

Fax: 812-855-1076

-----Original Message-----
From: Hites, Ronald A.
Sent: Monday, April 24, 2006 2:01 PM
To: Basu, Ilora
Subject: RE: IADN & LMLS

OK -- please tell Melissa what is going on.

Ron

-----Original Message-----
From: Basu, Ilora
Sent: Monday, April 24, 2006 1:32 PM
To: Hites, Ronald A.
Subject: RE: IADN & LMLS

Ron,
It looks like to me that she is talking about only the hard copies of the data. At one point, you and I talked about keeping our hard copies only for five years. Those must be tl data from 1990-1994. These are all reported and published. So I agree with you. That can be dumped.
But we need to inform Melissa before we do this.

Ilora

Ilora Basu

Research Scientist

School of Public and Environmental Affairs

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1315 E. 10th Street

Indiana University

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Bloomington, IN 47405

Phone: 812-855-2926 (off)
812-856-4364 (lab)

Fax: 812-855-1076

-----Original Message-----
From: Hites, Ronald A.
Sent: Monday, April 24, 2006 1:10 PM
To: Basu, Ilora
Subject: RE: IADN & LMLS

What do you recommend?

In my opinion, if it is just data, then let's have her dump everything.
If there are any samples or extracts, then let's have her send them to us.

Ron

-----Original Message-----
From: Basu, Ilora
Sent: Monday, April 24, 2006 1:05 PM
To: Hites, Ronald A.
Subject: FW: IADN & LMLS

Ron,
What do we do about it?

Ilora

Ilora Basu

Research Scientist

School of Public and Environmental Affairs

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812-856-4364 (lab)

Fax: 812-855-1076

-----Original Message-----
From: Harlin, Karen [mailto:kharlin@uiuc.edu]
Sent: Monday, April 24, 2006 12:35 PM
To: Basu, Ilora
Subject: IADN & LMLS

Hi Ilora, I hope you are well and enjoying life. How are you and your family? Are you still working? If so are you still working on IADN?

Its housekeeping time in the warehouse area and I have MANY boxes of IADN and LMLS data that were removed from offices, file cabinets and such. At one time EPA wanted me to send them to you for storage. Its been over 10 years. Let me know if you want them, because they need to be moved very soon. If you don't want them, then they'll go to the dumpster. If you want them, do you have a billing number for shipping?

Best Wishes,

Karen

Karen S. Harlin
Assistant Coordinator, National Atmospheric Deposition Program (NADP), Program Office
Director, Central Analytical Laboratory Illinois State Water Survey
2204 Griffith Dr.
Champaign, IL 61820
217-244-6413
217-244-3054
kharlin@uiuc.edu
http://nadp.sws.uiuc.edu/cal

**17.18: Reporting Met data only for sampling days**

Hi Ron and Ilora,

This makes a lot of sense to me, especially since we are now using more met data from other sources for our loading calculations. I approve of this suggestion.

Todd

"Hites, Ronald A." hitesr@indiana.edu
To: Todd Nettesheim/R5/USEPA/US@EPA
04/01/2009 11:19 AM
Cc: "Basu, Ilora" <ilora@indiana.edu>
Subject: FW: e-mail to Todd regarding met data

I fully concur with this suggestion – it would save us a lot of time and some money, both of which could be devoted to more productive activities.

Ron Hites

From: Basu, Ilora
Sent: Wednesday, April 01, 2009 11:06 AM
To: Hites, Ronald A.
Subject: e-mail to Todd regarding met data

Dear Todd,
Recently, Ron and I were discussing about the way we process and report our meteorological data. We are thinking of making little modification in the way we report it. Please let us know your thoughts about it.

**Our meteorological data files for each sampling site are very big. Our data logger collects data every 6 seconds and takes hourly average. We download data from data logger to our excel spreadsheet in the lab which ends up in 8787 rows and 12 columns. We check each and every data point to make sure that the sensors have worked properly. In case of sensors malfunctioning, we go to some other sites like NOAA or local airport to collect those bad or missing data. After going through this, we report the whole file for all 365 days (hourly averages) to the Data Base Manager.** But most of these data are not used. We only use the data for the sampling days to correlate them with the organic data.

This means that we are spending lot of time for something which are not useful. So we were wondering if we can report the met data of those sampling days only. In that case, instead of looking and correcting data for 365 days we will have to look at data from approx. 30 days.

Although we will download the whole 365 days data and store them properly in our spreadsheets as raw data. If necessary, these data will be available anytime . Please let us know your opinion.

Ilora

Ilora Basu
Research Scientist
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Phone: 812-855-2926 (off)
812-856-4364 (lab)

Fax: 812-855-1076

## 17.19. Communication on QA/QC issues

We have simplified the original requests to eliminate redundancies, included GLNPO's comments and questions in green font (a color we thought appropriate for the Agency), and added IU's responses.

IADN's CURRENT STATE:

Since IADN started more than 20 years ago, the levels of several target compounds have been steadily declining, proving that regulatory actions have had a measurable effect on the environment. The atmospheric concentrations of some of these chemicals are approaching levels below the sensitivities of our analytical instruments. Despite that trend, we continue to successfully measure more than 100 individual compounds in three different phases, and we have to date generated over 1,500,000 distinct measurements in about 10,000 samples since the beginning of the program. The most recent Quality Assurance Report, covering 2005-2012 (dated 31 December 2015) from Helena Dryfhout Clark, showed that we are still doing a very good job, and only a few issues were identified. We are working to find ways to address the fact that some chemicals are approaching the limits of detection. We think that doubling the sampling time is a viable option, and we are actively exploring this option.

We are also continuing to expand the suite of target compounds we measure to ensure that IADN is aligned with the EPA's vision. We are collecting preliminary data on HBCDD, bisphenol A, and long-chain perfluorinated chemicals, which are all included in the latest list of Chemicals of Concern. We have also started measuring a list of about 12 current-use pesticides.

**********

1) Request: We solicited GLNPO's comments on the "What about the damn blanks" paper.

   **GLNPO's Questions/Comments: Are you planning to resubmit the "What about the Damn Blanks" paper? Kindly re-state options and/or measures taken to reduce high blank values in PBDEs.**

   **IU's response: No, we are not planning to submit this paper to another journal – even with a more professional title. After much sole-searching, we have decided that this is too narrow an issue on which to base a scientific paper; thus, we plan to use this sort of analysis and exposition as part of the next paper we submit on the temporal and spatial trends of IADN analyte. We will also make additional efforts to deal with "non-detects" in our statistical calculations. The question is: Should a spreadsheet cell be left empty (our current approach) for non-detects, or should we use a more sophisticated statistical approach for dealing with left-censored data? We expect to work on this issue over the next year or so. In addition, we will continue our investigations at Eagle Harbor focused on increasing the sample to blank ratio for PBDEs by doubling our sampling time. Preliminary data are encouraging, but we need more information before making this fundamental change in our sampling operation procedures.**

**********

2) Request: We want to stop reporting BDE-7, -10, -30, -71, -119, -126, -138, -156+169, -180, -184, -191, -196, -204 and -205 in all samples at all sites. These fifteen PBDEs are detected less than 22% of time in vapor, particle, and precipitation samples. Put another way, none of these compounds were in the commercially produced PBDE mixtures at anything but trace levels. Removing these fifteen congeners will save time and effort in tracking their vanishingly small concentrations and in dealing with QA/QC issues.

**GLNPO's Response/Comments: We cross referenced and discussed these congeners within the context of GLNPO's Great Lakes Fish Monitoring Program with Beth Murphy. We are OK with Indiana University's proposal to stop reporting these 15 PBDE congeners. Kindly document this change as an addendum and modify the approved QAPP accordingly for submittal and review by EPA.**

**IU's response: Thank you. We will make these changes to the QAPP and send it for EPA's review.**

**********

3) Request: We want to drop the hexabromocyclododecanes (HBCDDs) from the flame retardant list. We know from the literature that these compounds cannot be measured accurately with GC/MS, which is our primary tool for the flame retardants measurements. We have been working with the Indiana University Mass Spectrometry Facility to develop an analytical method for the measurement of HBCDD isomers using liquid chromatography – mass spectrometry.

**GLNPO's Response/Question: We certainly empathized and do recognized the challenge and difficulty faced by Indiana University in analyzing HBCDD via GC-MS. Conversely, we fully support Indiana University's efforts to further investigate the feasibility of analyzing this chemical through other instrumentation methods such as HPLC-MS/MS. We feel that continued analyses and reporting of HBCDD is important since this chemical is designated as one of six "Chemicals of Mutual Concern." Is there a value or is it practical to continue analyzing HBCDD via GC-MS in tandem with LC-MS/MS?**

**IU's response: We do not think that it is necessary to continue analyzing HBCDD with both GC-MS and LC-MS/MS. GC-MS does not provide reliable HBCDD concentration measurements and is not able to separate the α-, β-, and γ-HBCDD isomers. Separation of these isomers is important in HBCDD measurements because it provides insight into its sources. We agree that HBCDD is an important analyte, and we will continue our efforts to develop an analytical method using HPLC-MS/MS on an instrument in IU's**

**Chemistry Department. At the moment, we have some preliminary data, which look promising.**

**********

4) Request: We want to stop measuring methoxychlor and β-HCH in all samples. The concentrations of these pesticides have never been large enough to accurately quantitate, and they contribute less than 5% to total pesticide concentrations in the samples. We would save a bit of time by not including these compounds in our reporting and QA/QC work.

GLNPO's Response/Comments: See comments on page 4 (now item #5) of this document. Kindly expand Table 2 (on page 14 of the Progress Report) to include % β-HCH relative to % Total HCH.

IU's response: Please see below the table showing percent contribution of β-HCH to total HCH at each site in vapor and particles.

**Table 1: β-HCH's average % contribution towards total HCH (for all years of measurement).**

| | Vapor | Particle | Precipitation |
|---|---|---|---|
| **Eagle Harbor** | **2.8** | **24.1*** | **12.3** |
| **Sleeping Bear Dunes** | **5.9** | **10.5*** | **17.4** |
| **Sturgeon Point** | **6.1** | **9.5*** | **18.7** |
| **Cleveland** | **17.2** | **51.0** | **18.7** |
| **Chicago** | **10.6** | **49.9** | **23.0** |
| **Point Petre** | **8.3** | **16.8*** | **21.2** |
| **Average** | **8.5** | **50.4** | **18.5** |

*Measurements stopped in 2003. These data cover 1997-2002.

Table 1 above shows that β-HCH contributes to roughly 50% of total HCH in the particle phase, about 20% in precipitation and about 9% in the vapor. The Quality Assurance Report from 31 December 2015 shows that the blank-to-sample ratios are generally 50% in the filters (measured only at Chicago and Cleveland) and ~30% in the vapor. Despite somewhat lower than desirable QA/QC performance, this compound is detected at relatively high frequency, so we withdraw our request to stop its measurement.

**Table 2: Methoxychlor average % contribution towards total pesticides (for all years of measurement).**

| | Va-por | Particle | Precipitation |
|---|---|---|---|
| **Eagle Harbor** | **0.9** | **10.6*** | **4.3** |
| **Sleeping Bear Dunes** | **1.3** | **7.6*** | **5.6** |
| **Sturgeon Point** | **1.1** | **6.6*** | **6.6** |
| **Cleveland** | **1.2** | **6.4** | **4.3** |
| **Chicago** | **1.4** | **7.0** | **4.3** |
| **Point Petre** | **1.0** | **6.3*** | **3.1** |
| **average** | **1.2** | **6.6** | **3.7** |

*Measurements stopped in 2003. These data cover 1997-2002.

Table 2 shows that methoxychlor contributes only 1% to total pesticides in the vapor phase, 7% in the particle phase, and 4% in precipitation. The Quality Assurance Report from 31 December 2015 shows that the blank-to-sample ratios are generally 20% in the particle phase filters (measured only at Chicago and Cleveland) and >100% in the vapor phase. In light of the low detection frequency, high sample-to-blank ratio, and poor gas chromatographic performance (this peak alone requires extensive instrument maintenance), we request to stop the measurements of methoxychlor in all phases.

**In summary, we confirm our request to stop measuring methoxychlor, but we will continue to measure β-HCH.**

**********

5) Request: We want to stop measuring all pesticides at Chicago and Cleveland in the particle phase. We stopped measuring these compounds in the particle phase at Eagle Harbor, Sleeping Bear Dunes, and Sturgeon Point on 1 January 2003. By now the concentrations of almost all of the pesticides in the particle phase, even at Chicago and Cleveland, are very low in the particle phase and rarely exceed our detection limits in these samples. On average, these concentrations do not exceed 1 pg/$m^3$ in both Chicago and Cleveland samples, and the overall sample to field blank ratio is 3:1. We would, of course, continue to measure all of these compounds in the vapor phase at all sites.

GLNPO's Response/Comments: Kindly expand Table 3 (on page 15 of the April-June 2017 Quarterly Report) to include pesticide levels (ng) in the vapor phase and perhaps provide visual graphs (e.g., bar graphs for Chicago and Cleveland sites) demonstrating how these chemical compounds are partitioning between particle and vapor phases to better assess how field blanks relative to organochlorine pesticides are changing over a time and in shorter intervals of maybe 5 and/or 10-year time periods.

**IU's response:** **Table 3 and Figure 1 below show partitioning of each pesticide (%) in vapor and particle at Chicago and Cleveland sites. Pesticides contributing to vapor concentrations > 30% are highlighted. Figure 2 shows sample-to-blank ratios for total pesticide concentrations in Chicago and Cleveland samples collected during 1996-2015 and 2003-2015, respectively. Please also see the attached Excel spreadsheet for more detailed data, including sample-to-blank ratios for each pesticide in both vapor and particle phases collected at Chicago (1996-2015) and Cleveland (2003-2015).**

**Table 3. Percent contributions towards the vapor + particle concentrations in Cleveland (2003-2015) and Chicago (1996-2015) samples.**

| | Cleveland | | Chicago | |
|---|---|---|---|---|
| | Particle | Vapor | Particle | Vapor |
| a-HCH | 3 | 97 | 1 | 99 |
| b-HCH | 29 | 71 | 37 | 63 |
| g-HCH | 11 | 89 | 7 | 93 |
| Heptachlor epoxide | 19 | 81 | 16 | 84 |
| Oxychlordane | 14 | 86 | 13 | 87 |
| g-Chlordane | 16 | 84 | 16 | 84 |
| Endosulfan I | 34 | 66 | 28 | 72 |
| a-Chlordane | 18 | 82 | 15 | 85 |
| t-Nonachlor | 7 | 93 | 10 | 90 |
| Dieldrin | 34 | 66 | 31 | 69 |
| o,p'-DDD | 49 | 51 | 48 | 52 |
| Endrin | 25 | 75 | 23 | 77 |
| Endosulfan II | 62 | 38 | 54 | 46 |
| p,p'-DDD | 69 | 31 | 50 | 50 |
| Endosulfan sulfate | 51 | 49 | 41 | 59 |
| p,p'-DDT | 17 | 83 | 22 | 78 |
| Methoxychlor | 69 | 31 | 64 | 36 |
| Hexachlorobenzene | 0 | 100 | 0 | 100 |
| p,p'-DDE | 0 | 100 | 0 | 100 |
| Aldrin | 0 | 100 | 0 | 100 |
| op-DDT | 0 | 100 | 0 | 100 |
| Octachlorostyrene | 0 | 100 | 0 | 100 |
| Σ DDTs | 12 | 88 | 12 | 88 |
| Σ Chlordanes | 14 | 86 | 14 | 86 |
| Σ Endosulfans | 35 | 65 | 29 | 71 |
| Σ HCHs | 8 | 92 | 5 | 95 |
| Σ Pesticides | 10 | 90 | 11 | 89 |

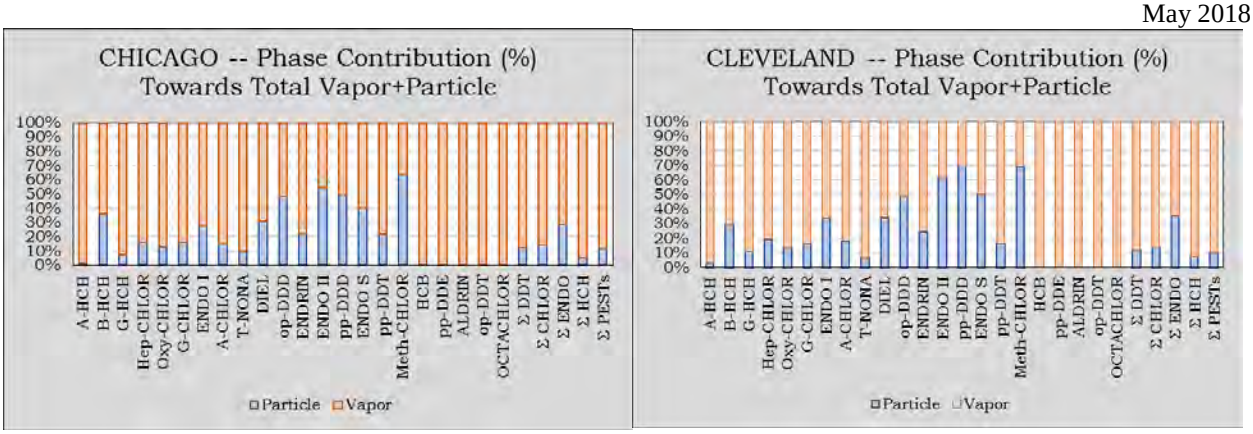


**Figure 1. Pesticides partitioning (%) between vapor and particle phases in Cleveland (A) and Chicago (B) samples.**

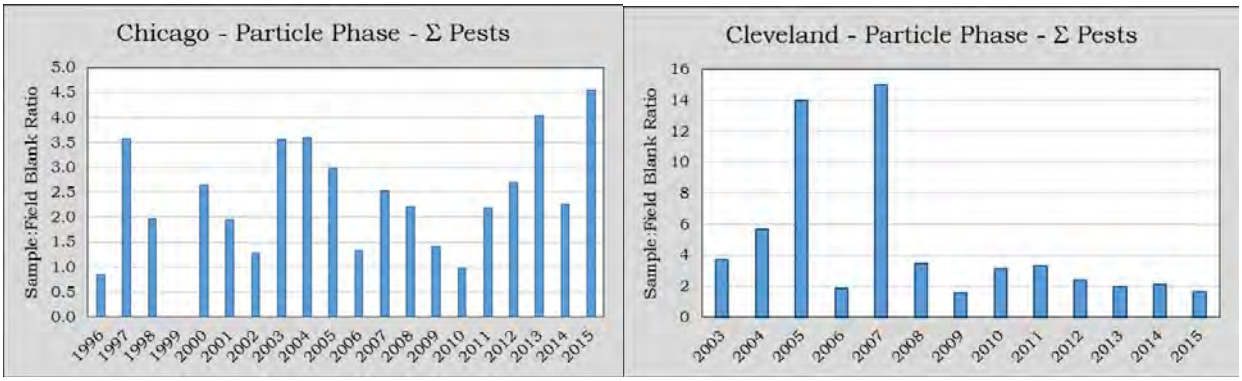


**Figure 2. Sample-to-blank ratios for total pesticide concentrations in particle phase samples collected at Chicago (1996-2015) and Cleveland (2003-2015).**

**Since there are 8 pesticides (see Table 2) for which the particle phase concentrations contribute more than 30% to total vapor + particle concentrations, we have decided to withdraw our request for now. We are currently looking into doubling the sampling time, which would improve the sample-to-blank ratios for these pesticides at these two sites.**

**********

6) Request: Although we have not been reporting it for some time, we want to remind everyone that we have an unexplained interference with PCB-44, and this congener should never be included in QA/QC analyses or in any data reports. The data for PCB-44 should be removed from the all data analyses in any reports. We do not report PCB-44 in our current data.

**GLNPO's Response/Comment: Duly noted.**

**IU's response: Thank you.**

**********

7) News item: The National Park Services set up a temporary weather station at our site at Sleeping Bear Dunes.

**GLNPO's Question: Is there something more we can do to further minimize and control contamination potentially posed by the temporary weather station set up by the National Park Service at the Sleeping Bear Dune site?**

**IU's response: The way the station is set up now it is unlikely it will cause contamination in IADN samples. However, we will keep monitoring field blanks from Sleeping Bear Dunes for potential contamination by the weather station.**

**********

8) Request: We have been asked to investigate high PAH blank values at Chicago for contamination: In Chicago, PAH blank-to-sample ratios range from 11-31% with an average of ~25%. The detection rates range from 52-73%. We have already taken or will soon be taking the following actions: (*a*) Both high-volume samplers at Chicago were thoroughly cleaned, and a new inlet throat and filter cassette will be installed in these samplers. (*b*) Test filter field blanks will be collected in each of these samplers. (c) In addition, potential sample storage room contamination will be investigated. The Chicago building is in poor condition, and the room where the sampling media is stored could be a potential source of contamination. Test filter blanks will be deployed in the room and in the desiccator used for storing the filters to pin point potential storage room contamination problems, if any.

**GLNPO's Questions: What applicable dataset (if any), were impacted or flagged as a result of high PAH blank values at the Chicago site due to contamination? What data corrective measures (in terms of data usability and reporting) were taken to correct for contamination found in the affected dataset?**

**IU's response: As it was summarized in the IADN QA/QC report (31 December 2015), PAH blank levels constituted around 25% of sample PAH levels (slightly higher than the desired 20%). Blank correcting may be considered in order to compensate for the elevated blank levels. We are currently investigating PAH contamination at Chicago site. We have sampled many blanks at Chicago since this problem has been identified, including blanks taken in the sample storage room and on the roof, and we think the problem is related to our samplers. We are now in the process of cleaning and replacing parts in the samplers in order to eliminate the contamination source.**

**********

9) Request: PCB levels at Eagle Harbor are decreasing and getting closer to the blank levels. The detection rates are low (some <10%) for the more highly chlorinated PCBs at Eagle Harbor, but most rates are still in the range of 70-90% for the less chlorinated PCBs. We will continue to measure PCB levels at Eagle Harbor. However, we may request to stop these measurements at this site in the future if there is a substantial decrease in the detection rates for the most of the target PCBs.

**GLNPO's Response/Comments: Due to the bi-national strategic importance of PCBs in the context of being designated as a "Chemicals of Mutual Concern" and also a "flagship" chemical compound analyzed by the IADN program for reporting purposes, we feel that PCBs merit further analyses.**

**GLNPO's Question/Recommendation: Would analysis of PCBs benefit from a longer sampling collection time (i.e., from 24 to 48 hours) to increase the sample signal? Our rationale for this line of thinking is, if we can somehow demonstrate that even after increasing the signal (and improving the accuracy of our measurement), a trend continues towards a decrease in PCBs concentration over time, say between a 5 and/or 10 - year interval, then, we definitely need to take a step back and reconsider whether IADN should continue monitoring PCBs in the future.**

**IU's response: As mentioned above, we are currently investigating the possibility of a longer sample collection time at Eagle Harbor (and perhaps at all other sites) with the purpose of producing better flame retardant measurements. We will, of course, include PCBs in this investigation in order to evaluate this possibility.**

**********

10) Request: We have been asked to investigate low recoveries for *p,p'*-DDT and *trans*-nonachlor for the organochlorine pesticide particulate measurements in urban air. Matrix spike data for pesticides in the particle phase were based on the recoveries from the analyses of the 50% hexane in DCM fraction only. However, part of *p,p'*-DDT and *trans*-nonachlor elutes in the 100% hexane fraction along with the PCBs. We do not analyze PCBs in filters; hence, the 100% hexane fractions for these matrix spike samples were not analyzed for the missing *p,p'*-DDT and *trans*-nonachlor that elute in the 100% hexane fraction. We suggest these data should be excluded from the report.

**GLNPO's Response/Comments: We concur with your assessment and findings. Kindly document this change as an addendum and modify the approved QAPP accordingly for submittal and review by EPA.**

**IU's response:** **Thank you. We will add these changes to the QAPP and send it for EPA's review.**

**********

11) Request: BDE-17, -28, -126, and -154 co-elute with other peaks present in the common reference standard, and the recoveries for these peaks should be adjusted using the sum of the concentrations of the target peak and of the co-eluting peak. We suggest repeating the duplicate sampling analyses separately for each site in order to pin-point which sites are doing less well in the duplicate sampling.

**GLNPO's Response/Comments:** **We concur with your assessment and findings. Kindly document this change as an addendum and modify the approved QAPP accordingly for submittal and review by EPA.**

**IU's response:** **Thank you. We will add these changes to the QAPP and send it for EPA's review.**

**********

12) Request: We suggest using a different method for the calculation of the limits of detection and blank-to-sample ratios. We think these calculations should be based on mass and not on concentrations.

**GLNPO's Response/Questions:** **Kindly submit to EPA proposed method for the calculation of LOD and blank to sample ratio calculations and re-iterate rationale for this change. Kindly clarify if this proposed mass change calculation is applicable only to precipitation samples or with all phases? How will this change affect past dataset that were calculated using volume instead of mass? Will there be a need to reconcile past dataset calculated by volume for reporting purposes?**

**IU's response:** **The limit of detection and blank-to-sample ratio calculation methods described above were used in the IADN QA/QC report (31 December 2015): That report says, "… the blank values from the analysis are divided by the average sample volume. For IU, the average air volume is 815 $m^3$, and for OAL it is 350 $m^3$. Then the ratio of the average blank to the average sample is calculated as: Blnk/Smpl = average blank concentration / average sample concentration".**

**We have always corrected for blanks and calculated blank-to-sample ratios based on mass and not on concentrations in our publications and blank reports. This is because the volume for blank samples is not measured in the field (the sampler is not turned on for blanks) and estimated as an average volume of 815 $m^3$. We are now looking into the advantages or disadvantages of both methods as well other possible approaches. Once we have found what we think is the best method, we will send you an update.**

**********

**Other items from Ron Hites:**

**We will submit a QA/QC report every year starting with 2016 data. This will include summaries of laboratory blank data, matrix spike and common reference standard recoveries, and the results of linearity and IDL tests.**

**As noted in your cover e-mail, we are at a bit of a loss regarding QA/QC management. In the old days, GLNPO had a QA/QC person (Lou Bloom, I think) who monitored and signed the QA/QC documents, read and approved the annual QA/QC reports, and interacted with IU and the Canadians to make sure everything was on track. I assume this person has moved on within the Agency or retired. As of last week, we received a notice that Helena Dryfhout-Clark of Environment Climate Change Canada (ECCC) has also stopped doing this this job.**

**In any case, this is a problem. What we really need is a neutral third party who will act as the project's QA/QC Officer. Having an employee of either ECCC or IU to this job would be a conflict of interest. At the moment Marta Venier, who is a co-director of IADN at IU, signed the most recent IU QAPP. This is not right. I don't know who, if anyone, has signed the Canadian equivalent document.**

**In 2000-2005, we had a contractor named Peter Fowlie who was paid by a grant from GLNPO to ECCC and was IADN's QA/QC officer. In my opinion this worked well. He operated as a neutral third party to make sure the QA/QC procedures were documented and working. Funds for his position disappeared a few years ago, and he was never replaced.**

**We really need to find someone, preferably not an EPA or ECCC employee and certainly not an IU employee, who would act as the QA/QC officer on a more or less permanent basis. I know funds are an issue, but it seems to me that this position is getting to be more and more essential to maintain the project's integrity.**

## 17.20. Summary of All Changes:

| Changes | Dates |
|---|---|
| 1. Metal Analysis stopped | January 2001 |
| 2. TSP/TOC stopped | August 1996 |
| 3. PBDE started | January 2003 (method development) |
| 4. MIC wipe stopped | June 1999 |
| 5. Filter composite stopped | December 1996 |
| 6. HCB, P,P'-DDE stopped on filter composite | September 1996 |
| 7. Pesticide Analysis on filters, Chicago, and Cleveland continued | May 2003 |
| 8. Aerochem stopped | March 1995 |
| 9. Belfort Rain gauge stopped | August 2004 |
| 10. MIC collection day changed from 28 days to one month | August 2004 |
| 11. Changes in PCB standard | January 2005 |
| 12. Common Reference Standard distributed by Peter Fowlie | 2004 |
| 13. Indoor Temperature recording Chart stopped | January 2006 |
| 14. PCB analysis stopped in EH, SBD, and in STP rain samples | June 2006 ( from January 2006 samples) |
| 15. Reduction in QC samples (FB, FD, LD, LB, MS) | July 1996 |
| 16. Reduction in Field Trip | December 1998 |
| 17. Stopping of Performance standard | December 2005 |
| 18. Stopping of Temperature Recorder | January 2006 |
| 19. Stopping PCB Analysis in Precipitation Samples of Eagle Harbor, | June 2006 |
| 20. Reduction of FB and FD from 10-5% | July 2006 |
| 21. Moving MIC-2 from Eagle harbor to Chicago | July 2006 |
| 22. Common Reference Standard Batch 2, by Celine Audette | June 2006 |
| 23. 6890 installed | June 1999, and March 2005 |
| 24. 1701 analysis started | 2004 samples |
| 25. Epsilon HCH started as surrogate standard for 50% fraction | **July 2007** |
| 26. Point Petre duplicate sampler closed | September 2007 |
| 27. Meteorological sensors installed at Cleveland | **April 2009** |
| 28. Meteorological data processed only for sampling days. | April 2009 |
| 29. GC 5890 closed | October 2009 |
| 30. Moved to Multidisciplinary Science Building II | October 2009 |
| 31. XAD and Sodium sulfate drying procedure changed | February 2010 |
| 32. XAD drying procedure | January 2010 |
| 33. PBDE CRS started | March 2010 |
| 34. IADN Visualization tool release | March 2017 |

**17.21** Memorandum of Understanding (MOU)

**INDIANA UNIVERSITY**

*MEMORANDUM OF UNDERSTANDING*

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**TO: Marta Venier**
**FROM: Allen Tucker**
**University Information Technology Services**
**DATE: 8/12/2016**
**SUBJECT: SMART Client Memorandum of Understanding**

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**Thank you again for the opportunity to serve your technology needs during the upcoming fiscal year. It is a privilege for University Information Technology Services (UITS) to serve the university community and provide a meaningful service to our clients. We take this responsibility seriously and have dedicated ourselves to ensuring the highest possible support and quality available at this university.**

**This Memorandum of Understanding (MOU) is an agreement between the Indiana University Information Technology Services, herein after called 'UITS', and The School of Public and Environmental Affairs (BL-SPEA), herein referred to as the 'Customer' for University delivered SMART Services.**

**Purpose**

**This MOU is intended to define the relationship between UITS and the Customer with respect to providing SMART Administration services for the Customer managed system, including service offering description, service rates, scope of UITS responsibilities, and scope of customer responsibilities.**

**Service Description**

**SMART services is a multi-level support service offering that provides systems management of a server. The SMART service offers 'basic' and 'standard' support levels. These two levels of support can be customized with 'add-on' services that augment base levels of service. The support options for SMART Services include:**

**Base Level Services**:

**All levels include 24/7 system outage support and business hours general support requests.**

**Basic Support**

a. Basic support includes all Operating System support (patching, upgrades, and security).
b. In this option, the customer's IT Professional manages all issues or configurations that are not associated with the operating system or underlying hardware or infrastructure.

**Standard Support**

c. Standard support includes all Operating System support (patching, upgrades, and security), as well as additional support of Active Directory group/OU/GPO management, file share permission support, IU IT Policy and security alignment reporting and review.
d. Standard support expands the level of management responsibility into a more comprehensive level of support provided by UITS.

Add-On Services:

**Database hosting / administration**

- Paired with Basic Support, this option allows customers to take advantage of UITS Database Administrator expertise to host, manage, and maintain Oracle, MySQL, or MS SQL databases.

**3rd Party application hosting / administration & vendor management**

- Paired with Standard Support, this option allows customers to take advantage of UITS Systems Administrator expertise to host, manage, and maintain 3rd party applications.
- In this option, if issues arise that are associated with the operating system OR the application, all communication, troubleshooting, and vendor management is coordinated through UITS.

**IU IT Policy and security alignment reporting**

- Can be paired with basic or standard support
- System resource dashboard made available to customer.
- Reports to include patching, UISO vulnerability, anti-malware, IT-12, backup

**Performance monitoring and reporting**

- Can be paired with basic or standard support
- System resource dashboard made available to customer.
- Dashboards to include CPU, memory, disk, uptime

**Service Responsibilities**

**All service responsibilities for UITS and the Customer are outlined in detail in Appendix B of this document.**

**Service Rates**

**The SMART Administrative Services rates and fee provided below are subject to change annually, effective every July 1st, with the start of each new University fiscal year. In the unlikely event of a necessary, but unexpected rate modification outside the annual renewal period, the Customer will be given 60 days' notice prior to the adjustment. It should also be noted that SMART Administrative Services are billed monthly in arrears and are *in addition* to the base cost of Intelligent Infrastructure server and disk resources.**

**The SMART Administrative Services monthly rate and fee schedule for the annual term of this MOU and agreed to by UITS and the Customer is as follows:**

## Base Level Services

| | |
|---|---|
| **Basic Support** | **$119** |
| **Standard Support** | **$357** |

## Add-on Services

| | |
|---|---|
| **Database Hosting / Administration [1]** | **$179** |
| **3rd Party Application Hosting / Administration & Vendor Management [2]** | **$139** |
| **Policy and security alignment dashboards available to local IT Pro [3]** | **$29** |
| **Performance monitoring and dashboards available to local IT Pro [4]** | **$29** |

***All prices are subject to change***
***A one-time set up fee of $952 will be charged for all new server builds***

***[1] Service only available with Basic Support***
***[2] Service only available with Standard Support***
***[3] Reports to include patching, UISO vulnerability, anti-malware, IT-12, backup***
***[4] Dashboards to include CPU, memory, disk, uptime***

**(As the computing needs of each Customer may vary across time, an individualized quotation will be created and submitted for Customer agreement and approval according to the schedules noted above.)**

**Account Information**
**The following Customer details and account information are to be utilized for the instantiation and utilization of UITS SMART Administrative Services.**

**University Account Number: _____4339905_____**

☐ **University/Departmental funded**

☑ **Grant funded**

☐ **Joint funded (University/Departmental and Grant funded)**

**If grant funding is to be used for this project, please indicate the grant expiration date: 10/31/2019_____.**

**In the event of a required Customer account number change due to account expiration, retirement or the like, the Customer will provide UITS a replacement account number within 60 days or request formal termination of the service. UITS reserves the right to suspend activity for SMART Administration Services having invalid or expired University account numbers.**

**Effective Date and Signature**

**This MOU shall be effective upon the signature of UITS and Customer authorized representatives. It shall be in effect until such as time either party terminates the agreement and will automatically renew on an annual basis.**

**As part of this agreement, and with the beginning of each fiscal year, the customer will be provided an updated Smart Administration Services Addendum outlining any modifications in service levels, including updated rate and fee schedules.**

**UITS and the customer indicate agreement with this MOU by their signatures.**

*Allen E. Tucker /s* **5/25/2018**

**Allen Tucker** **Date**
**Manager, HELPnet Central Systems**

**5/25/2018**

**Marta Venier** **Date**
**School of Public and Environmental Affairs (BL-SPEA)**

**Statement of Work: BL-SPEA–** SMART-00009_1

**The below costs are estimates and are subject to change if the environment is expanded or reconfigured.**

**From**
**SMART Services**

**Estimate For**
**BL-SPEA**

| | |
|---|---|
| **Estimate ID** | **SMART-00009_2** |
| **Issue Date** | **08/9/2016** |
| **Subject** | **Estimate for SMART Services** |

| Item Type | Description | Quantity | Unit Price | Amount |
|---|---|---|---|---|
| **Add-On Service** | **Initial OS Configuration, Firewall Config, II Request, Backup Config, etc** | **1.00** | **$952.00** | **$952.00** |
| **Support Level** | **Standard Support - One new build (application server)** | **1.00** | **$357.00** | **$357.00** |
| **Add-On Service** | **3rd Party Application Hosting / Administration & Vendor Management** | **1.00** | **$139.00** | **139.00** |

| | |
|---|---|
| **Subtotal** | **$1,448.00** |
| **UITS Admin Fee (6.50%)** | **$94.12** |
| **Estimate Total** | **$1,542.12** |

**Notes**
**All Intelligent Infrastructure costs will be directly billed to the customer and are not included in this estimate. The estimated costs for this specific environment are as follows: 1 Application server – 2 x CPU, 4 GB RAM, 60 GB Disk, TSM Backup = $52.75 / month.**

*Appendix A.*

**SMART Services: Service Level Expectations**

**Related policies**

- Security of Information Technology Resources (IT-12)
- Cyber Risk Mitigation (IT-28)

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## Contents

**1.) General overview**

**University Information Technology Services (UITS) provides systems and database administration services to the Indiana University campuses. This Service Level Expectation (SLE) is specific to the service known as the SMART Services.**

**This is an SLE between SMART Services Customers and UITS. The scope of this document includes:**

- **Services provided by UITS to SMART Services Customers**
- **Levels of response time, availability, and support associated with these services**
- **Responsibilities of the UITS service provider and responsibilities of the Customer**
- **Processes for requesting services and getting support**

**This SLE covers the period from July 1, 2016 to June 30, 2017, and will be reviewed and revised at the end of this period.**

**OR**

**This SLE shall remain valid until revised or terminated.**

1.1 Terms and definitions

- **Backup solutions: Optional service available for subscription that provides cross-site backups and cross-campus failover options, which isolate you from potential disasters by securing your backup data within hardened data centers**
- **Business day: Normal working day in the time zone where Indiana Data Center facilities are located (Eastern Time Zone -5 GMT and participates in Daylight Savings Time)**
- **Customer: The party identified as the engaging organization to this Agreement with UITS**
- **Designated contacts: Customer-named contacts, technical resources, and fiscal account resources which are established, person-specific email addresses associated with the Customer support contract. It is expected that these contacts will be updated upon any personnel or responsibility change by the Customer.**
- **Intelligent Infrastructure (II): Virtual Server Hosting Services, where unlike "co-location" or other physical server hosting services, a virtual server is leased and the Customer is not required to make an initial investment in buying capital equipment such as servers and storage hardware. On-call support coverage for service outages is 24 hours a day, 7 days a week, 365 days a year.**
- **SMART Services: A multi-level systems administration support service offering that provides systems management of a server. The SMART service offers 'basic' and 'standard' support levels. These two levels of support can be customized with 'add-on' services that augment base levels of service. On-call support coverage for service outages is 24 hours a day, 7 days a week, 365 days a year.**
- **Problem resolution: The use of reasonable commercial efforts to resolve the reported problem. These methods may include (but are not limited to) configuration changes, patches that fix an issue, replacing failed hardware, reinstalling software, etc.**
- **Respond: Addressing the initial request and taking ownership of the issue**
- **Response time: The amount of time elapsed between the initial contact by the Customer to UITS and the returned response to the Customer by UITS staff**

- **Service Level Expectation (SLE): The Customer Service Level Expectation (SLE) that identifies the features and defines the processes involved with the delivery by UITS of various support functions to Customers, as presented by this document's content**
- **Service Request (SR): A single issue opened with UITS. The SR number identifies the Service Request.**

- **Severity Definitions for SMART Services:**
  - **Severity 1 (Urgent):**
    a. **An error with a direct security impact on the service**
    b. **An error isolated to the Service Infrastructure production environment that renders the Service Infrastructure inoperative or causes the Service Infrastructure to fail catastrophically; i.e., critical system impact, system down**
    c. **A reported defect in the production environment, which cannot be reasonably circumvented, in which there is an emergency condition that significantly restricts the use of the product to perform necessary business functions**
    d. **Inability to use the product or critical impact on operation requiring an immediate solution.**
  - **Severity 2 (High):**
    a. **An error isolated to the Service Infrastructure that substantially degrades the performance of the service or materially restricts business; i.e., major system impact, temporary system hanging**
    b. **A reported defect in the Service Infrastructure, which restricts the use of one or more features of the Service Infrastructure to perform necessary business functions but does not completely restrict the use of the Service Infrastructure**
    c. **Ability to use the Service Infrastructure, but an important function is not available, and operations are severely impacted**
  - **Severity 3 (Medium):**
    a. **An error isolated to the Service Infrastructure that causes only a moderate impact on the use of the service: i.e., moderate system impact, performance/operational impact**
    b. **A reported defect in the Service Infrastructure that restricts the use of one or more features of the Service Infrastructure to perform necessary business functions, while the defect can be easily circumvented**
    c. **An error that can cause some functional restrictions but does not have a critical or severe impact on operations**
  - **Severity 4 (Low):**
    a. **A reported anomaly in the Service Infrastructure environment that does not substantially restrict the use of one or more features of the Service Infrastructure to perform necessary business functions; a minor problem and not significant to operations**
    b. **An anomaly that may be easily circumvented or may need to be submitted to UITS as an enhancement request**
- **UITS: University Information Technology Services, which is staffed by professional support personnel providing assistance with diagnosis and resolution of defects and/or failures in II services**
- **Service Infrastructure: The method to supply the software and server infrastructure and network capacity necessary to host the SMART Services service.**
- **Workaround: A change in the environment or data to avoid error without substantially impairing use of the II service**

## 2.) Service descriptions

2.1 Service scope

**SMART services is a multi-level support service offering that provides systems management of a server. The SMART service offers 'basic' and 'standard' support levels. These two levels of support can be customized with 'add-on' services that augment base levels of service. The support options for SMART Services include:**

### 2.1.1 Base Level Services:

**All levels include 24/7 system outage support and business hours general support requests.**

2.1.1.1 Basic Support

a. Basic support includes all Operating System support (patching, upgrades, and security).
b. In this option, the customer's IT Professional manages all issues or configurations that are not associated with the operating system or underlying hardware or infrastructure.

2.1.1.2 Standard Support

a. Standard support includes all Operating System support (patching, upgrades, and security), as well as additional support of Active Directory group/OU/GPO management, file share permission support, IU IT Policy and security alignment reporting and review.
b. Standard support expands the level of management responsibility into a more comprehensive level of support.
c. In this option, the customer's IT Professional manages all issues or configurations that are not associated with the operating system or underlying hardware or infrastructure.

### 2.1.2 Add-On Services:

2.1.2.1 Database hosting / administration

a. Paired with Basic Support, this option allows customers to take advantage of UITS Database Administrator expertise to host, manage, and maintain Oracle, MySQL, or MS SQL databases.

2.1.2.2 3rd Party application hosting / administration & vendor management

a. Paired with Standard Support, this option allows customers to take advantage of UITS Systems Administrator expertise to host, manage, and maintain 3rd party applications.
b. In this option, if issues arise that are associated with the operating system OR the application, all communication, troubleshooting, and vendor management is coordinated through UITS.

2.1.2.3 IU IT Policy and security alignment reporting

a. Can be paired with basic or standard support
b. System resource dashboard made available to customer.
c. Reports to include patching, UISO vulnerability, anti-malware, IT-12, backup

2.1.2.4 Performance monitoring and reporting

a. Can be paired with basic or standard support
b. System resource dashboard made available to customer.
c. Dashboards to include CPU, memory, disk, uptime

### 2.1.3 Service exclusions

**Service does not include the following items or actions:**

**a. On-site services or support**
**b. Modifications of software code, security-policy configuration, audits, or security design on Customer systems**

**UITS shall have no obligation to support:**

**a. Problems caused by Customer negligence, misuse, misapplication, or use of the product beyond the control of the UITS**

**b. Operating systems that are past their end-of-support date as listed by the operating system vendor**

**c. Products installed, intentionally or unintentionally, that result in nefarious activities**

2.2 IU Data Centers IUB and IUPUI

**IU has two hardened data centers, one each on the Bloomington and Indianapolis campuses. The Data Centers provide a safe and secure location for IT equipment. This includes the basic infrastructure of standardized cabinets and cabinet distribution units for power. Additionally, the Data Centers have uninterruptible power supplies (UPS), power distribution, and HVAC to provide year-round cooling and protect equipment from environmental hazards of dust, temperature, and humidity. Diesel generators will provide ongoing power in the event of a campus or Data Center power outage. Enhanced cabinet power distribution provides redundant circuits and remote monitoring of the power distribution. Physical security includes proximity card readers and biometric hand scanners for access authentication, ID cards, reinforced doors, security glass, and alarms. Fire suppression equipment is provided by a double inter-lock pre-action sprinkler system. Additionally, both facilities have UITS staff on site in the building 24 hours a day, 7 days a week.**

2.3 Operating parameters

**Trained operators provide support for the Data Center 24 hours a day, 7 days a week. Operations staff monitor vital data center and server information. Examples include: temperature, network connectivity, and server vitals as set up by the Systems Administrator. Problem coordination/management, notification, escalation, and reporting are done by the operations staff.**

2.4 System level

**Certified Systems Administrators provision, configure, and maintain the Service Infrastructure to allow the Customer to utilize the SMART Services service. Provisioning includes the setup and configuration of the operating system, server side configuration files, firewall rules, and server networking interoperability. Ongoing support includes upgrades, monitoring, performance tuning, expansion, and software patches of the servers hosting the service infrastructure.**

## 3.) Customer Roles and Responsibilities

3.1 Customer obligations

**Customer responsibilities and/or requirements include:**

- **Staffing: All Customer personnel contacting UITS for support must be fully trained on the operating system running in the Service Infrastructure.**
- **Named designated contacts:**
  - **Customer-named contacts, technical resources, and fiscal account resources, which are established, person-specific email addresses associated with the Customer support contract. It is expected that these contacts will be updated upon any personnel or responsibility change.**
  - **Customer Active Directory Services (ADS) group to be used for Service Infrastructure resource assignment. The ADS group contents are managed by the Customer, thereby providing the most control over resource access to the Customer.**
- **Full responsibility for system administration: System administration falls into, but is not limited to, the following areas:**
  - **Data management, as prescribed by university policies and state and federal laws and regulations in respect to protection of, access to, and confidentiality of institutional or personal data residing on or processed by the system**
  - **Liaison or manager who will provide operations staff with support escalation and contact information for system administration functions. Contact information for billing and operational inquiries.**

- **Data protection is solely the responsibility of the Customer.**
- **Active incident response plan as outlined by University Information Policy Office (UIPO)**
- **Managing local system logs for operating system and application-related troubleshooting**
- **Regular scheduled auditing for abnormal events including intrusion detection**
- **If production systems are deemed critical, test VMs should be installed and maintained. In the event an issue is discovered, the test VM can be utilized to test the fix quickly and deploy it with confidence in the production environment.**
- **In the event that critical data is stored within log data sent to SMART Services, the Customer is responsible for securing data approval from the Committee of Data Stewards. The environment is managed to standards that allow for storage of critical data.**
- **University Information Technology policy IT-12 requires that university organizational units manage technology resources securely.**

### 3.1.1 System administration

- **At the Customer system level, log review, performance, system status, resource usage, and events that may result in security issues and identify any required performance tuning.**
- **Maintain base system and network security. This includes system and application patching, firewall settings, and associated infrastructure components of the systems.**

### 3.1.2 Backup and removal of data

- System logs should continue to be maintained and stored on local system per UISO/UIPO duration guidelines.
- **To reconstruct lost or altered Customer data, Customers must maintain a separate backup system on the Customer managed system that is not dependent on the software or hardware products under support. Note: Optional backup services are offered by UITS Storage and Virtualization that provide cross-site backups and cross-campus failover options, which isolate potential disasters by securing backup data within hardened Data Centers. The Customer is responsible for backup contents; UITS is responsible for maintaining media services to host the backup content.**

### 3.1.3 Security and privacy

- Technicians may access and use system-generated logs and other content-neutral data describing the use of technology resources for the purposes of analyzing system and storage utilization, problem troubleshooting, security administration, and in support of audits.
- Technicians may not disclose specific information technology resources assigned to, or electronic information associated with, an individual except as authorized under Policy IT-07 Section 1.
- Technicians may access and use system-generated logs and other content-neutral data describing the use of technology resources for systems and services which they are authorized.
- Logs, events and other content-neutral information describing the use of technology resources may be exported, extracted, copied, etc., from the service only when necessary for the purposes outlined above. In these cases, users of this data must ensure that they are securely stored. This data must be securely destroyed as soon as they are no longer needed.

3.2 Service provider requirements

**UITS responsibilities and/or requirements include:**

### 3.2.1 Charges (if applicable)

**Customer billing for services will occur monthly.**

### 3.2.2 Assumptions

- **Services are clearly documented on the IU UITS Knowledge Base.**
- **Major upgrades will be treated as a project outside the scope of this document.**
- **Changes to services will be communicated and documented.**

### 3.2.3 Hardware and infrastructure technology updates

- **The physical compute resources serving the SMART Services workload are hosted on high-end enterprise-class x86 hardware. The x86 hardware has an expected lifecycle replacement of approximately every 36 months. The replacement process may require the Service Infrastructure to be momentarily power cycled to complete the migration process. The migration process can be scheduled during normal Customer maintenance activities at the convenience of the Customer.**
- **The storage supporting the SMART Services environment is hosted on high-end enterprise-class SAN. The SAN lifecycle replacement occurs between 48 and 60 months. The replacement process does require Service Infrastructures to be interrupted.**
- **Patch processing for the x86 hardware, hypervisor, and SAN occur concurrently and do not require Service Infrastructures to be interrupted during updates.**
- **UITS will provide adequate hardware for both x86 compute and storage required to support the Customer workload.**

### 3.2.4 Physical hardware - system administration

- **At the service infrastructure level, review logs and performance counters to obtain system status required to identify and correct potential server or software problems.**
- **Apply critical patches as recommended for the environment.**
- **Perform system tuning as needed to the server environment.**
- **Assign client settings and manage access.**
- **Coordinate with vendors for any maintenance or support requests.**
- **Capacity planning for physical resources (servers, storage, load balancers)**

### 3.2.5 Backup and removal of data

- **Upon termination of services, the Service Infrastructure and data will be securely erased in accordance with IU IT policies and procedures. All programs and data that were served via the SMART Services offering will no longer be accessible.**

### 3.2.6 Problem determination

- **Coordinate with the vendor for any required support.**
- **Determine if the problem is hardware, software, or storage by reviewing the system event logs.**

### 3.2.7 Backups/storage of backups

- **The SMART Services service utilizes the backup services provided by the UITS Storage and Virtualization Team. Virtual server rental includes an off-site backup of Customer system images and Customer operating systems; data volumes are excluded from this protection service, but can be backed up via Tivoli Storage Manager (TSM).**
- **Tivoli Storage Manager (TSM) software is used for backups in conjunction with a virtual server rental to backup system files and configuration that are critical to the restoration of the service upon catastrophic failure of the service infrastructure.**
- **Data backups will occur for all service infrastructure by installing a TSM backup agent on the virtual server. A backup of the Customer server is run every night, 365 days a year. TSM stores the current version and up to two old versions of each file. This also includes an off-site copy of the data.**

### 3.2.8 Network services

**Act as a liaison to UITS Network Engineering for problem reports and incident handling.**

### 3.2.9 Security and privacy guidance

- Systems administrators may access and use system-generated logs and other content-neutral data describing the use of technology resources for the purposes of analyzing system and storage utilization, problem troubleshooting, security administration, and in support of audits.
- Access and use of system-generated logs and other content-neutral data describing the use of technology resources beyond these purposes requires authorization under Policy IT-07 Section 1.
- Maintainers will ensure that logs and other content-neutral data describing the use of technology resources older than 60 days are removed from the log collection and analysis service. Maintainers will ensure that all purged logs and backup files are securely destroyed.
- Incident Response staff and UISO Engineers may use system-generated, content-neutral information for the purposes of investigating reports of abuse or misuse of university information technology resources and in support of audits.
- Incident Response staff and UISO Engineers may not disclose or permit access to specific information technology resources assigned to, or electronic information associated with, an individual except as authorized under Policy IT-07 Section 1.
- System-generated logs and other content-neutral data describing the use of technology resources used in investigating reports of abuse or misuse of university information technology resources or in support of audits are exempt from the retention rule. System-generated logs and other content-neutral data describing the use of technology resources will be stored and disposed of in a secure manner.

**4.) Hours of coverage, support, response times, and escalation**

4.1 Hours of system administration support

**The Service Infrastructure request queue for support requests is monitored Monday-Friday 8 am – 5 pm, with the exception of university holidays.**

4.2 Service requests

**The process to request utilization of the service requires submission via the SMART Services Service Request Form on the on the SMART Services website. In support of services outlined in this document, UITS will respond to service-related incidents and/or change requests submitted by the Customer through Service Infrastructure request.**

**Note: DO NOT submit a service request for a Severity 1 issue via the web request form. For a Severity 1 case, contact UITS directly by telephone (812-855-9910), and request that a Severity 1 incident be opened with the SMART administrators group related to SMART Services. An incident number will be generated and sent to the Customer via email. Provide and include any additional details that may be relevant to the case.**

4.3 Service request priorities and response times

| Priority | Criteria | Example | Target response time* |
|---|---|---|---|
| **Low- Severity 4** | **a. Service Infrastructure environment that does not substantially restrict the use of one or more features of the Service Infrastructure to perform necessary business functions; this is a minor problem and is not significant to operations**<br>**b. An anomaly that may be easily circumvented or may need to be submitted to UITS as an enhancement request** | **I would like to onboard a new server, how do I accomplish this?** | **UITS and Customer will provide resources during normal business hours for problem resolution.** |
| **Medium- Severity 3** | **a. An error isolated to the Service Infrastructure that causes only a moderate impact on the use of the service: i.e., moderate system impact, performance/operational impact**<br>**b. A reported defect in the Service Infrastructure that restricts the use of one or more features of the Service Infrastructure to perform necessary business functions, while the defect can be easily circumvented**<br>**c. An error that can cause some functional restrictions but does not have a critical or severe impact on operations.** | **Service Infrastructure occasionally hangs on while opening application. Please help research a resolution.** | **UITS and the Customer will commit full-time resources during normal business hours for problem resolution to obtain a workaround or reduce the severity of the error and alternative resources during normal business hours.** |

***Target response time is defined as the time between receipt of the call and the time that a Support Team member begins working on the problem. Due to the wide diversity of problems that can occur, and the methods needed to resolve them, response time IS NOT defined as the time between the receipt**

**of a call and problem resolution. UITS does not guarantee the resolution of a problem within the times specified.**

### 4.3.1 Normal incident processing

**In the event that a Customer accidentally or incorrectly assigns a request priority, UITS will correct the priority by utilizing the severity definitions. Communication with the Customer will occur for any priority change.**

**Service providers supporting this service will prioritize incoming service incidents as normal priority unless the service incident fits one or more of the criteria listed in the major incident handling section of this document.**

**When an IT Request ticket is opened for a Customer via the web interface:**

- **The Support Center will respond to the Customer and process all new IT Request tickets within 8 business hours.**
- **Low (Severity 4) priority incidents will be resolved within 30 days with a status provided every five days.**
- **Medium (Severity 3) priority incidents will be resolved within five days with a daily status provided.**

### 4.3.2 Major incident handling

**UITS staff supporting this service will prioritize an incoming incident request as high priority if it meets any one of the following criteria:**

- **Significant number of people affected**
- **Organizational structure is a multiplier for number of people affected**
- **Percentage of total tasks that can no longer be performed by individuals**
- **Academic and Administrative Calendar deadlines**
- **Significant impact on the delivery of instruction**
- **Significant or lasting impact on student academic performance**
- **Significant risk to law, rule, or policy compliance**

**Urgent (Severity 1) priority incidents will be resolved within eight business hours with a status provided every two hours.**

**High (Severity 2) priority incidents will be resolved within one day with a status provided every six hours.**

**The infrastructure is protected and supported by vendor support 7 days a week, 24 hours per day. If incidents are linked to vendor related components, an appropriate level support case will be opened with the vendor. The Customer will be updated by SMART Services staff with case progress.**

#### 4.3.2.1 Service request priorities and response times

| Priority | Criteria | Example | Target response time* |
|---|---|---|---|
| **High- Severity 2** | **a. An error isolated to the Service Infrastructure that sub-** | **The allocated memory on the server is being** | **UITS and the Customer must commit full-time resources during non-standard business hours for problem resolution, to** |

| Priority | Criteria | Example | Target response time* |
|---|---|---|---|
| | **stantially degrades the performance of the service or materially restricts business; i.e. major system impact, temporary system hanging**<br>**b. A reported defect in the Service Infrastructure which restricts the use of one or more features required to perform necessary business functions. While the error does not completely restrict the use of the Service Infrastructure, operations are severely impacted.** | **fully allocated and thus causing service degradation.** | **obtain workaround, or reduce the severity of the error.** |
| **Urgent- Severity 1** | **a. An error with a direct security impact on the service**<br>**b. An error isolated to the Service Infrastructure production environment that renders the Service Infrastructure inoperative or causes the Service Infrastructure to fail catastrophically; i.e., critical system impact, system down**<br>**c. A reported defect in the production environment which cannot be reasonably circumvented, in which there is an emergency condition that significantly restricts the use of the product to perform necessary business functions**<br>**d. Inability to use the product or critical impact on operation requiring an immediate solution** | **Server is not functional; network is not available; service is unavailable.** | **UITS and the Customer must commit the necessary resources around the clock for problem resolution to obtain workaround or reduce the severity of the error. UITS will use commercially reasonable efforts to make services available with a monthly uptime percentage of at least 99.9% during any monthly billing cycle.** |

***Target response time is defined as the time between receipt of the call and the time that a Support Team member begins working on the problem. Due to the wide diversity of problems that can occur and the methods needed to resolve them, response time IS NOT defined as the time between the receipt of a call and problem resolution. UITS does not guarantee the resolution of a problem within the times specified.**

4.3.2.2 Major incident response times

| Service provider | Service hours and conditions | Backup contacted under what conditions | Escalation rules | Response time from notification |
|---|---|---|---|---|
| **SAV** | **24/7** | **SMART Services service performance degradation** | **Follow on-call contact list for off hours and normal senior management escalation** | **1 hour** |
| **Data Center Operations** | **24/7** | | **Follow on-call contact list for off hours and normal senior management escalation** | **5 minutes** |

4.4 Maintenance management

### 4.4.1 Service maintenance/change management

**The SMART Services infrastructure services adhere to the UITS Change Management process. Service providers for this service adhere to the UITS Maintenance Window Guidelines. Review the UITS Scheduled Maintenance windows.**

**All services and/or related components require regularly scheduled maintenance ("maintenance window") in order to meet established service levels. These activities may render systems and/or applications unavailable for normal user interaction.**

**Due to the technology available within the virtual infrastructure, a maintenance window is not reserved for II. Patches are implemented to the infrastructure in a rolling mode, which ensures Service Infrastructures are available during the infrastructure maintenance. UITS will use commercially reasonable efforts to make SMART Services available with a monthly uptime percentage of at least 99.9% of the time in a given month.**

### 4.4.2 General exceptions to the standard maintenance window

| Exceptions | Parameters | Coverage |
|---|---|---|
| **University holidays** | | **Unless emergency or 24/7 supported servers** |
| **Fiscal year close** | **Last business day in June** | **Unless emergency** |
| **Finals/grade weeks** | | **Unless emergency** |

## 5.) Reporting, reviewing, and auditing

**IU Internal Audit performs periodic audits of the SMART Services. This document should be reviewed a minimum of once per fiscal year. However, in lieu of a review during any period specified, the current document will remain in effect.**

5.1 Term and termination

- **Term: Support shall be provided in annual terms and shall be renewable to then-current support plan when UITS is notified of Customer's intent to renew the existing contract, or UITS is notified of Customer's intent not to renew services.**
- **Termination: Customer may terminate this service via submission of a support request. Services are billed in arrears based on actual usage; charges will be processed through month of service termination.**

  **Upon termination of services, Service Infrastructure and data will be securely erased in accordance with IT policies and procedures. All programs and data that were served via the II offering will no longer be accessible.**

5.2 Service Level Expectation (SLE)

**SLE Update: This agreement and related UITS plan offering details are operational in nature and may be modified any time by UITS. UITS will communicate in advance proposed changes to Customer. The Customer may terminate the Customer relationship without penalty if all parties cannot abide by the revisions. This agreement supersedes any previous service level expectation.**

5.3 Miscellaneous

**Force Majeure: Except for the obligation to pay monies due and owing, neither party shall be liable for any delay or failure in performance due to an event outside the defaulting party's reasonable control, including without limitation, acts of God, earthquakes, labor disputes, shortages of supplies, actions of governmental entities, riots, war, fire, epidemics, or other circumstances beyond its reasonable control. The obligations and rights of the excused party shall be extended on a day-to-day basis for the period equal to the period of the excusable delay.**

*Appendix B.*

**Service Responsibilities**

| Customer Responsibility | Responsibility Description |
|---|---|
| **Funding** | **A fiscal account contact and account number needs to be established prior to the beginning of any services. It is expected that upon any changes to either account number or account contact will be communicated to Smart Administration Services.** |
| **Designated Contacts / Technical Contacts** | **The customer must designate at least one individual as contact for all matters related to the use of this service. SMART or Computer Operations will only act upon requests made by or approved by the designated contact(s). A list of designated customer personnel with contact information (e-mail and phone numbers) will need to be provided to Smart Administration Services. It is expected that these contacts will be updated upon any personnel or responsibility changes.**<br>**These contacts will assist with Smart service instantiation, maintenance and problem resolutions.** |
| **Active Directory Services (ADS) Group** | **An ADS group needs to be requested, created and maintained by the customer. This group will be used to grant user access to the server.** |
| **VPN Group** | **A VPN group needs to be requested and maintained by the customer. This VPN group will be used to grant computer/workstation access to the server(s).** |
| **Root / Admin Access** | **The customer can request root / admin access for their technical staff and/or vendors for their environment for application installation and troubleshooting. These individuals are expected to apply IU "Best Practices" as outlined by UISO and UIPO for server access and authentication.**<br>**If for any reason a system becomes unusable do to customer or vendor misconfiguration, Smart Administration Services will only assist by offering to restore the server back to the most recent DR snapshot.** |
| **Application End User Support** | **The customer is responsible for providing the application end user support.** |
| **Access to the Environment** | **The customer is completely responsible for controlling access to their application and workspace.** |

| | |
|---|---|
| **Data Management & Security** | **Data management and security, as prescribed by university policies and state and federal laws and regulations in respect to protection of, access to, and confidentiality of institutional or personal data residing on or processed by the system, is the responsibility of the customer.**<br>**It is the customer's responsibility to make sure the following information is not stored anywhere in your workspace without approval of University Counsel, Internal Audit, Data Stewards, University Information Technology Security and University Information Policy Office.**<br>**This includes but is not limited to:**<br>• **Social Security Numbers**<br>• **Credit card numbers**<br>• **Financial account numbers**<br>• **Debit card numbers**<br>• **Security codes, access codes and passwords**<br>• **Driver's license numbers**<br>• **State identification card numbers**<br>• **HIPAA Regulated Data** |
| **Application End User Support** | **The customer is responsible for providing the application end user support.** |
| **Application Security** | **The customer is responsible for applying appropriate industries data security practices and responsibilities in accordance with UISO and UIPO policy.**<br>**The customer is responsible for having their application scanned by the UISO application scanner and for any intervention/resolution required.** |

| | |
|---|---|
| **Vendors and Vended Applications** | **If a third-party vendor is involved with the delivery of the system, the customer is responsible for:**<br>• **Providing an ongoing Vendor relationship and a support point of contact with the Vendor for issue resolution.**<br>• **Providing Vendor oversight monitoring and accountability should Vender require direct server access for the installation, maintenance and issue resolution of the software system.**<br>• **Providing technical support to assist Vendor in configuring and customizing the software solution to meet priority and functionality needs.**<br>• **Providing end user support and management of the application with assistance from the Vendor.**<br>• **Providing, with the assistance from the Vendor, the acquisition and support of all non-university standard third-party software required by said system.**<br>• **Ensuring Vendor installs and configures said system on operating and database system builds provided by UITS.**<br>• **Ensuring the Vendor will provide maintenance services with respect to said system.**<br>• **Ensuring the vended application version or revision levels remain current with supported operating system and database versions and upgrades.**<br>• **Ensuring the Vendor will provide appropriate application security practices in accordance to UISO policy and HIPAA Security Rule controls by said systems.**<br>• **Ensuring access to the Vendor support services for off-hours technical support during regular software upgrades on a mutually agreed upon pre-arranged basis with the Customer and UITS for said system.**<br>• **Ensuring access to the Vendor support services for assistance in resolving issues relating to hosting environment in support of said system** |

*Appendix C.*

**Technical Responsibility Matrix**

**Operating System Installation and Configuration**

| Description | RHEL (6.X) | Windows 2012 R2 (64-bit) | Service Notes |
|---|---|---|---|
| Assist in defining, documenting, recommending and reviewing environment architecture | ✓ | ✓ | |
| Assist in defining, documenting, recommending and reviewing VM build specifications | ✓ | ✓ | |
| Install Operating System | ✓ | ✓ | To IU standards and best practices. |
| Apply current OS security patches | ✓ | ✓ | Windows patches for all non-database servers are applied weekly as applicable, identified by IU WSUS and determined by Microsoft.<br><br>Linux patches for all non-database servers are applied weekly as applicable as identified by the IU RHEL repository. |
| Install and setup up local host and Network (NOC) firewalls | ✓ | ✓ | To IU standards and best practices. |
| Request and obtain server IP and DNS | ✓ | ✓ | |
| Request, evaluate and resolve issues relating to initial UISO host security scan | ✓ | ✓ | |
| Install and patch any Windows or Linux OS products, as required by the customer | ✓ | ✓ | Windows products part of the OS roles and features including but not limited to:<br>· File Services<br>· Print Services<br>· Remote Desktop Services<br>· IIS<br>· .Net Framework<br>· Microsoft Office Products<br>Linux products within the IU RHEL repository. |

**Backups**

| Description | RHEL (6.X) | Windows 2012 R2 (64-bit) | Service Notes |
|---|---|---|---|
| Installation and maintenance of TSM Client | ✓ | ✓ | |
| Provide setup of cross-site, replicated backups | ✓ | ✓ | |
| Monitoring of nightly backup and issue resolution | ✓ | ✓ | Resolution to be jointly completed by the Customer and Enterprise Infrastructure. |

**Systems Administration and Ongoing Maintenance**

| Description | RHEL (6.X) | Windows 2012 R2 (64-bit) | Service Notes |
|---|---|---|---|
| Ongoing maintenance of Operating System | ✓ | ✓ | |
| Perform annual DR recovery testing | ✓ | ✓ | As requested by Customer. |
| Monitor server logs and resolve errors impacting system availability. | ✓ | ✓ | Resolution to be jointly completed by the Customer and UITS. |
| 1Provide Production System Troubleshooting and Critical Issue[1] Resolution | ✓ | ✓ | All Hours with 1 hour response. |
| Provide Production/Test/Development Troubleshooting and *Non-Critical* Issue Resolution | ✓ | ✓ | Normal Business Hours (8 am to 5 pm), with 4 hour response subject to normal issue submission and resolution procedures. |
| Provide Server OS Level Performance Tuning | ✓ | ✓ | Memory, I/O, CPU, etc. |
| Provide a way to submit requests for assistance via IU Footprints ticket system. | ✓ | ✓ | |

**Security**

| Description | RHEL (6.X) | Windows 2012 R2 (64-bit) | Service Notes |
|---|---|---|---|
| Application of Operating System patches and upgrades | ✓ | ✓ | Adhere to current UITS standard patching schedule. |
| Review and evaluation of monthly scheduled UISO host security scans | ✓ | ✓ | Resolution to be jointly completed by the Customer and UITS |
| Account Management | ✓ | ✓ | Completed in partnership with Account Management Team and Customer. This only pertains to server access. Application access is the responsibility of the customer. |
| Datacenter Firewall and Host-Based Firewall Configuration | ✓ | ✓ | |
| Configuring Encryption Technologies | ✓ | ✓ | Completed in partnership with UITS and Customer, per customer requirements. |
| Installation and setup of antivirus software | ✓ | ✓ | As requested by customer and to IU standards and best practices. |
| Request and installation of SSL certificates | ✓ | ✓ | |

[1] **Critical Issue is defined as a production system which in unavailable, unusable or presenting data privacy concerns.**

| Description | Oracle | MSSQL 2012/2014 | MySQL | Service Notes |
|---|---|---|---|---|
| Define, Document, Recommend and Review Database Installation Specifications. | ✓ | ✓ | ✓ | |
| Install RDBMS according to Vendor Specifications | ✓ | ✓ | ✓ | |
| Configure RDBMS according to Installation Specifications | ✓ | ✓ | ✓ | |
| Validate RDBMS connectivity and Operation. | ✓ | ✓ | ✓ | |
| Performance monitoring, collection, and reporting with Solarwinds Database Performance Analyzer | ✓ | ✓ | ✓ | Monitoring includes: System Availability, Processor Utilization, Physical Memory and Storage Capacity. |
| Install applicable RDBMS Software and Patches | ✓ | ✓ | ✓ | |

**Database Installation**

**Database Administration and Ongoing Maintenance**

| Description | Oracle | MSSQL 2012/2014 | MySQL | Service Notes |
|---|---|---|---|---|
| Create, Configure and Schedule RDBMS Backup Scripts, per Customer Specifications. | ✓ | ✓ | ✓ | |
| Perform Annual Recovery Testing | ✓ | ✓ | ✓ | As requested by the Customer. |
| Monitor RDBMS System Logs and resolve critical errors impacting system availability. | ✓ | ✓ | ✓ | |
| Monitor and provide consultation regarding non-critical messages or errors with the RDBMS System. | ✓ | ✓ | ✓ | Resolution to be jointly completed by the Customer and UITS EI – DBA. |
| 2Provide Production System Troubleshooting and Critical Issue [2] Resolution | ✓ | ✓ | ✓ | All Hours with 1 hour response. |
| Provide Production/Test/Development Troubleshooting and *Non-Critical* Issue Resolution | ✓ | ✓ | ✓ | Normal Business Hours (8 am to 5 pm), with 4 hour response subject to normal issue submission and resolution procedures. |
| Perform RDBMS Vendor Management Activities, including creation of Service Requests, License Management, etc. | ✓ | ✓ | ✓ | |
| Server Level Performance Tuning | ✓ | ✓ | ✓ | Memory, I/O, CPU, etc. |

[2] **Critical Issue is defined as a production system which in unavailable, unusable or presenting data privacy concerns.**

| | | | | |
|---|---|---|---|---|
| **Database Cloning and Data Migration for the support of Systems Development.** | ✓ | ✓ | ✓ | **For Development/Test/Production Systems.** |

**Database Security**

| Description | Oracle | MSSQL 2012/2014 | MySQL | Service Notes |
|---|---|---|---|---|
| **Install applicable RDBMS Software Upgrades and Patches** | ✓ | ✓ | ✓ | **During Standard, Scheduled Maintenance Windows.** |
| **Account Management** | ✓ | ✓ | ✓ | **Completed in partnership with Account Management Team** |
| **Network Based Access Filtering** | ✓ | | | **Commonly known as 'IP Filtering'** |
| **Configuring RDBMS Encryption Technologies** | ✓ | ✓ | ✓ | **Network and Data Encryption, per customer requirements.** |
| **Maintain central repository and connection file template for Client Connectivity.** | ✓ | | | **Centralized TNS Names and Oracle Internet Directory** |
| **Long-term Secure Storage** | ✓ | ✓ | ✓ | **We use the Scholarly Data Archive for secure storage of large online logical backups as determined by the customer for HIPAA or project requirements** |

**Additional Database Services**

| Description | Oracle | MSSQL 2012/2014 | MySQL | Service Notes |
|---|---|---|---|---|
| **Application Performance Tuning (SQL Based Query Tuning)** | ✓ | ✓ | ✓ | **Ultimately, the Application Service Owner is responsible for the design and performance of SQL executed against the RDBMS. The Enterprise Database Administration team can assist in these activities, particularly in the identification of long running queries, but cannot be responsible for performance over which we have no design or control.** |
| **Automated Shell Script Development and Schedule Advising** | ✓ | ✓ | ✓ | **(CRON, BRTE.)** |
| **Data Modeling and Design. (Best Practices)** | ✓ | ✓ | ✓ | |
| **RDBMS Procedure Development (Functions, Procedures, etc.)** | ✓ | ✓ | ✓ | |
| **Client Connectivity Software (Application Server)** | ✓ | | ✓ | **Includes the Oracle Client, JDBC/ODBC connectivity and Cobol Precompilers, MySQL Connectors.** |

## 17.22 DHS Scan Authorization Letter

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Washington, D.C. 20460

To: Corporate Officer
3rd Party Hosting Provider Address

From: Robert McKinney
Senior Agency Information Security Officer (SAISO)
United States Environmental Protection Agency

Subject: Vulnerability Scan Authorization

Reference: Office of Management and Budget Memo M-15-01. Issued October 3, 2014 (https://www.whitehouse.gov/sites/default/files/omb/memoranda/2015/m-15-01.pdf)

Background
The Executive Office of the President (EOP) issued the referenced document in part to introduce new requirements for a proactive vulnerability scanning process to search for and potentially identify technical risks to US Government (USG) data. This applies to data regardless of physical location. With regards to data outside the EPA's boundary, this includes (and is specifically mentioned in Section 2 of this memo) vendors who manage, host or provide security for internet accessible systems, including external websites and servers.

The Department of Homeland Security (DHS) is identified as conducting the vulnerability scans. However, information from DHS indicates that actual letters of authorization are to be executed between the 3rd Party Vendor (3PV) and the Agency who owns the data – in this case the Environmental Protection Agency (EPA). These letters are to be provided to DHS with a requirement for semi-annual reviews and updates forwarded as necessary.

Purpose
The purpose of this letter is to provide authorization for DHS to perform periodic vulnerability scans against all servers or systems (hereafter "Systems"). While specific roles and responsibilities are outlined below, it should be understood that both signatories of this document are legally capable to make such an authorization.

Scope
The scope of this authorization includes the list of all internet accessible addresses and systems, including static IP addresses for external websites, servers and other access points and domain name service names for dynamically provisioned systems that store, process, transmit, or receive data on behalf of the EPA.

Vulnerability Scanning General Information

Vulnerability scans are attempts to discover and enumerate technical risks to computer systems and the data that is processed, stored or transferred on a given system. Scans can be conducted from "inside" an organization's network (internal scanning) as well as from outside the boundary of the organization (external scanning). This letter of authorization is for external scanning only, indicating that DHS will not need physical access to your facility for these activities. All scanning will take place over the Internet.

Vulnerability scans can be conducted in two ways: Credentialed or Non-Credentialed.

- Credentialed scanning involves the person conducting the scans to utilize a username and password (usually at administrator level but not always) in the scanning configuration. While this does provide for a deeper examination of the targeted systems configuration, most malicious actors do not possess these credentials. The DHS scans will be performed with the same limitation – no credentials – to mimic what malicious actors are able to identify over the Internet;
- Non-Credentialed scanning, as alluded to above, is the most common type of scanning activity that occurs over the Internet and is the method that will be used by DHS. No credentials will need to be issued as part of these engagements.

Vulnerability scanning involves actively probing computer systems for technical flaws, misconfigurations, etc. that malicious actors can potentially exploit in their mission. While every precaution is taken, it is possible for scanning activity to be disruptive to the targeted systems operation – from minor resource usage (CPU cycles, etc.) up to, in some cases, system shutdown or re-booting. Communications between the 3PV, EPA, and DHS is critical in the event any type of disruption negatively impacts the 3PV operation. **The DHS, EPA, and the US Government are not responsible or accountable for any loss of system(s), its resources, or any other contractual responsibilities between the 3PV and other customers.**

Roles and Responsibilities

Within the scope of this authorization letter and the referenced document, specific roles and responsibilities are as follows:

- 3PV
  - Provide a legally binding signature on this letter of authorization between 3PV and the EPA;
    - Perform an annual review of this authorization, making appropriate notifications as necessary;
  - Provide full contact information for a technical resource(s) that are the liaison between the 3PV, EPA, and DHS. Full contact information is defined as:
    - Name and Title;
    - E-mail address for daytime communications. If scanning is to take place after normal business hours, then a second e-mail address is required;
    - Daytime office phone number (if different from mobile number);
    - Daytime mobile number;
    - After Hours phone number(s) in the event scanning is to take place after normal business hours (local to the 3PV);

- A list of all internet accessible addresses and systems, including static IP addresses for external websites, servers and other access points and domain name service names for dynamically provisioned systems that store, process, transmit or receive data for or on behalf of the EPA;
  - Provide at least 5 days of advance notice to the EPA and DHS of changes to this list;
- Coordinate with the EPA and DHS remediation of vulnerabilities reported;

- EPA
  - Provide a legally binding signature on this letter of authorization between 3PV and the EPA;
  - Act as the liaison between the 3PV, the EPA, and DHS;
  - Provide a copy of this document, once signed by all parties, to DHS for their files.
    - Perform an annual review of this authorization, making appropriate notifications as necessary;
  - Provide to DHS:
    - The complete list of target identification information, i.e. IP addresses;
    - Technical Point of Contact (3PV-POC) information of the 3PV for communication in the event of a disruption to a scan target;
  - Distribute scanning results to the following:
    - 3PV-Point of Contact;
  - Coordinate annual review of this authorization letter;
  - Distribute any changes to the scan target list to DHS;
  - Coordinate remediation efforts and information between all affected parties;
- DHS
  - Coordinate logistics of vulnerability scans with 3PV-POC, including an emergency contact number in the event of system disruptions;
  - Perform vulnerability scans as required and within the scope of this authorization;
  - Distribute copies of vulnerability scan results to:
    - 3PV-POC;
    - EPA SAISO
  - Collaborate with 3PV-POC and EPA SAISO on remediation efforts to include, but not limited to, identification of false positives;

Signatures

Once signed and dated by both parties this letter of authorization shall be in effect for one year which shall include an annual review as required.

_____ Date: 25 Apr 15
Robert McKinney
US Environmental Protection Agency
Senior Agency Information Security Officer

_____ Date: _____
3rd Party Official
Name of Company
Title

### 17.23. Distribution List:

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