ANALYTICAL CHEMISTRY SERVICES

Background

Battelle is the world's largest nonprofit research and development organization with over 22,000 employees at more than 130 locations globally. A 501(c)(3) charitable trust, Battelle was founded on industrialist Gordon Battelle's vision that business and scientific interests can go hand-in-hand as forces for positive change. Today, Battelle manages the world's leading national laboratories and maintains a contract research portfolio spanning consumer and industrial, energy and environment, health and pharmaceutical, and national security research.

We are valued by our clients for our independence and ability to innovate within virtually any business or research climate. From large government agencies and multi-national corporations to small start-ups and incubator projects, Battelle provides the resources, brainpower, and flexibility to fulfill our clients' needs. Battelle's own mission includes a strong charitable commitment to community development and education. That's why we support staff volunteer efforts; science, technology, engineering and mathematics (STEM) education programs; and philanthropic projects in the communities we serve.

Battelle's Analytical Chemistry Services (ACS) group has a long history of providing high-quality analytical services to a variety of government and industrial clients. ACS has three major focus areas:

1. ***The fate and transport of potentially toxic and hazardous chemicals in the environment.*** Our environmental fate and transport research emphasizes detection of environmental contaminants at ultra-low levels, analytical methods development, and chemical fate of contaminants. Battelle specializes in the development of analytical chemistry methods for the measurement of trace organic compounds in complex environmental matrices and the conduct of programs designed to address fate, effects, and consequences of contaminants in diverse ecosystems. Research addresses contaminant types and amounts; the chemical transformations that occur to contaminants in the water column, soils, sediments, plants, and animals; and the biological factors and responses to these contaminants that affect bioavailability.

2. ***Consumer product testing.*** Our consumer products team's main focus is the Battelle World Detergent Program (BWDP), a multi-client subscription-level program that performs full chemical characterization on over 250 detergent, dishwashing soap, and hard-surface cleaner samples from around the world. The program, originally started at Battelle Geneva and transferred to Battelle Duxbury in 2009, has been providing high-quality data to clients for over 22 years.

3. ***Petroleum hydrocarbon forensics.*** For over 25 years, Battelle has been at the forefront of petroleum forensics. Since the early days of Exxon Valdez to ongoing work for other industrial clients, the saturated hydrocarbon, volatile hydrocarbon, polycyclic aromatic hydrocarbon, and petroleum biomarker analyses performed here have been employed by forensic scientists to determine the source of spills and assess the toxicity of petroleum releases. Many sample preparation and analytical techniques related to petroleum forensics were developed in the laboratories in Duxbury and are still commonly used today.

Battelle provides high-quality, cost-effective chemistry consulting and analytical laboratory services that provide clients with:

- Laboratory staff with over 30 years of experience in providing ultra-trace measurement of chemicals in all environmental media of importance
- Experienced project chemists with advanced degrees in environmental chemistry to design and oversee projects and represent our customers
- Codified methods with ultra-low detection limits that meet the most demanding risk-based analysis programs
- Significant laboratory and staff capacity to handle large projects in a timely and cost-effective manner
- A national reputation with regulators, industry, and state and local governments for providing the highest-quality analytical chemistry support for high-profile investigations
- Laboratory services for non-routine analyses and method development for unusual chemicals or media that cannot be addressed through the use of standard U.S. EPA or similar analytical protocols
- A reporting system tailored to meet work product formats specified by clients.

An integral part of Battelle's mission is technology transfer demonstrated through our training courses and seminars, and technology transfer support for laboratory design and implementation.

Analytical Laboratories and Analyses

The combination of laboratory facilities equipped with state-of-the-art instrumentation and analytical equipment coupled with our staff's scientific expertise in developing and applying sample preparation and analysis procedures gives Battelle's ACS Laboratory the ability to deliver robust, sensitive, and defensible data to our clients.

Battelle offers laboratory instrumentation networked through automated data acquisition and reduction systems, supported by a newly upgraded Laboratory Information Management System (LIMS). Effective information management is the key to successful laboratory operation, and the use of an automated information management system is essential to provide accurate and timely results and manage large laboratory projects. Battelle's LIMS automates laboratory data handling and report preparation, linking all aspects of laboratory operation from sample login to reporting and electronic data deliverable (EDD) preparation, providing effective laboratory management tools and advanced information automation capabilities for the entire laboratory process.

Sample preparation laboratories at Battelle are specially designed for ultra-trace analytical work (e.g., positive pressure and/or filtered incoming air, as appropriate) and have also been configured for high sample throughput. Separate sample preparation and sample storage areas are maintained to isolate high-level samples from samples requiring ultra-low processing and storage. All sample extraction and processing methods used by our laboratory are fully documented in Battelle's Standard Operating Procedures (SOPs). Separate analytical instrument rooms are dedicated to various types of gas chromatography (GC) analysis (e.g., ECD, FID); gas chromatography/mass spectrometry (GC/MS) analysis; purge and trap gas chromatography/mass spectrometry (P&T GC/MS); and high-performance liquid-chromatography (HPLC) and liquid chromatography tandem mass spectrometry (LC-MS/MS) analyses. The majority of our GCs have multiple detector configurations (e.g., dual detection and injection capabilities) for either simultaneous confirmatory analysis or increased capacity.

Our scientists often take standard published methods and refine and adapt them into our own SOPs to achieve optimal sensitivity when analyzing complicated matrices with multiple factors that can cause analytical interferences, such as industrial products, various biota sample and tissue types, or marine sediment and salt water. We are constantly revisiting and updating our SOPs to ensure that we keep abreast with changes in instrumentation and new ideas. A list of our routinely used analytical method references can be found in Appendix A.

Method Development and Validation

Along with comprehensive sample and data analysis capabilities, Battelle offers extensive capabilities in the development, evaluation, and application of new methods and technologies. Method development includes environmental sample collection, processing, analysis, and data interpretation for non-routine analytes and/or unusual matrices. Our analytical chemists have developed a number of methods with commonly employed analytical tools, such as GC/MS, GC/ECD, GC/FID, IC, and LC-MS/MS. Examples include but are not limited to:

- Analysis of oil dispersant components (dipropylene glycol n-butyl ether and dioctyl sodium sulfosuccinate) in water using GC/MS and/or direct-inject LC-MS/MS
- Analysis of glycols in water by direct-inject GC/FID and LC-MS/MS
- "Whole oil" analysis of crude oil and petroleum distillates fuel via direct injection of product using GC/MS
- Analysis of methyl ester sulfonate (MES) and secondary alkane sulfonate (SAS) surfactants in consumer products by LC-MS/MS.

The ACS group also has access to other more unique instrumentation, advanced analytical techniques, and expertise throughout the organization. Battelle staff have developed a number of advanced methodologies using analytical tools such as GC×GC/ToF-MS, GC/IRMS, and GC/PFPD. Examples include but are not limited to:

- Compound specific isotopic analysis (CSIA) of natural gas alkanes in water and gas and crude oil alkanes in oil by GC/IRMS
- Analysis of oil, WAF, and tarball samples by comprehensive gas chromatography time of flight mass spectrometry (GC×GC/ToF-MS)
- Analysis of the sulfur fingerprint of oil samples by gas chromatography pulsed flame photometric detection (GC/PFPD).

Quality

Battelle's ACS Laboratory is committed to providing the highest-quality analytical data to meet the needs of our clients and to ensuring that all environmental data collection activities are complete, representative, comparable, and of a known and documented quality. It is Battelle's policy that all field and laboratory data include, where possible, documented quality control (QC) data. To ensure data quality, quality assurance (QA) procedures are employed for all data-generating activities, from study design and sample analysis to data generation, reduction, and reporting. The Quality System is designed to ensure that the quality of data generated at the Laboratory meets or exceeds the needs of Battelle's ACS clients.

Battelle's QA program is presented in a site QA Manual (QAM), which describes the application of Battelle's Quality Management System within the ACS Laboratory. This manual summarizes laboratory policies and procedures for environmental analytical chemistry.

The Quality System elements in the QAM define policy requirements so that consistent technical management and data collection activities are implemented for each client. Both laboratory-wide and project-specific QC procedures are implemented for each project. At the laboratory level, QC procedures include rigorous staff training in each laboratory procedure that includes initial and ongoing demonstrations of capability; routine instrument calibration checks and maintenance to ensure that acceptance criteria are achieved prior to sample analysis; biannual performance evaluation (PE) samples; sample custody tracking throughout sample receipt, processing, analysis, and reporting via the LIMS; QC samples incorporated into each batch of samples processed for analysis; and control charts by matrix, method, and parameter. At the project level, QC procedures include assignment of a chemistry project manager; development of a Quality Assurance Project Plan that defines the project requirements, methods, QC samples, and reporting requirements; project-specific analytical batches so that QC samples are specific to each client's requirements; and a three-tiered QC review process including verification and validation by the analyst, QC chemist, and project manager.

Lab Accreditations

The ACS Laboratory is accredited by National Environmental Laboratory Accreditation Program (NELAP), which is based on ISO 9001 and ISO 17025. The QAM and detailed SOPs

NELAP Certification (Florida #E87856)			
	Water	Sediment	Tissue
PAH (GC/MS)	X	X	X
Pesticides (GC/ECD)	X	X	X
PCB Congeners (GC/ECD)	X	X	X
PCB Congeners (GC/MS)	X	X	X
n-alkanes (GC/FID)	X	X	

referenced throughout the QAM detail the activities and evaluation criteria necessary to ensure that analytical data generated by the ACS Laboratory meet NELAP requirements.

Project Experience

Battelle's ACS Laboratory stands behind its work, from sample receipt though project implementation and product delivery. We track customer satisfaction through our client survey process and follow up with action items to maintain continuous improvement. Below are some examples of successful projects that have resulted in repeat customer business spanning multiple years and contract awards.

Client: U.S. Army Corps of Engineers (USACE), New York District
Scope: Sampling and testing of dredged material

The New York District of the USACE selected Battelle as its prime contractor for providing analytical services in support of dredge material evaluation studies conducted as part of District dredging operations. Under the task order agreement, Battelle is providing the full spectrum of analytical chemistry services required for *Green Book* testing, including field sampling; preparation of conventional and modified elutriates; and analysis of sediment, site water, elutriate, and biological tissue for trace metals, polycyclic aromatic hydrocarbons (PAHs), chlorinated pesticides and polychlorinated biphenyl (PCB) congeners, dioxin and furans, and organotin compounds. Detection limits achieved for these analyses are typically 10 to 100 times lower than conventional U.S. EPA methods of analysis while meeting the demanding data quality objectives of *Green Book*. Battelle is the only analytical laboratory in the country with a Quality Assurance Project Plan compliant with the *Green Book* and the *Regional Testing Manual* approved and on file with EPA Region 2.

Client: U.S. EPA
Scope: Assessing the effectiveness of dredging activities at contaminated sediment sites

Battelle is currently supporting the U.S. EPA Office of Research and Development and Great Lakes National Program Office in their joint mission to determine remedy effectiveness at multiple sites within the United States, including the Ashtabula and Ottawa Rivers in Ohio, Lake Hartwell in Georgia, the Grand Calumet River in Indiana, the Kinnickinnic River in Wisconsin, and Ruddiman Creek in Michigan. This program seeks to combine physical, chemical, and biological data to determine the effectiveness of dredging as a remedial alternative. Battelle works with U.S. EPA to monitor macrobenthos and indigenous fish; deploys, retrieves, and analyzes surrogate sampling devices, such as semipermeable membrane devices (SPMDs) and solid-phase microextraction (SPMEs) fibers; monitors bathymetric change and sediment chemistry (surface and vertical profile); and monitors water quality prior to, during, and after dredging. In all, between 2006 and 2013, Battelle analyzed more than 5,000 sediments, waters, tissues, SPMDs, and SPMEs in support of these and similar projects for a wide range of chemicals, including an extended list of PCB congeners, PAHs, and alkylated-PAH compounds.

Client: An industrial oil & gas corporation
Scope: Provide defensible quality analytical chemistry and environmental forensic services for various programs

Beginning in May of 2010, Battelle has been providing high-quality, hydrocarbon fingerprinting analysis related to an environmental oil release.

Battelle continues to provide PAH, biomarker, SHC, TPH, and PIANO analytical results to the client, their consultants, and third party validators in direct support of this project.

To date, Battelle has analyzed nearly 10,000 samples of varying matrices (sediment, water, biological tissue, and oil) and reported data with turnaround times ranging from 24 hours to 10 days.

Client: Multi-client industry
Scope: Analytical chemistry-based compositional analysis on commercial detergents

The BWDP is a multi-client detergent research program. The original program, established in 1987, focused on Europe. Other global regions of interest to our clients were added in subsequent years: North America in 1990, Asia and Asian-Pacific Rim countries in 1996, and Latin America in 1997. Since 2009, industry has shown an increasing interest in Middle Eastern and African markets. Products from these geographic regions have been added to the study in response to the industry interest upon indication from clients. As a multi-client study, the analysis costs are spread across all subscribers.

Battelle's ACS Group performs analysis products annually and primarily focuses on laundry and dishwashing detergent products and hard-surface cleaners. The study quantitatively determines all major and minor ingredients so that the composition of the detergent may be reconstructed in weight percent. The analyses include such ingredients as:

- Surfactants
- Builders and fillers
- Bleaching systems
- Polymers
- Enzymes
- Complexing agents
- Foam control agents
- Hydrotropes
- Brighteners.

Appendix A – Analytical Methods

Compound Class	Sample Extraction Battelle SOP	Sample Extraction Description	Base EPA Analysis Methods Sample Analysis Battelle SOP
Semi-volatiles, Hopanes, Triterpanes, and Steranes	Tissue (SOP 5-190) Tissue (SOP 5-307) Tissue (SOP 5-309) Sediment (SOP 5-192) Sediment (SOP 5-307) Sediment (SOP 5-309) Water (SOP 5-200) Products/NAPL (SOP 5-334)	Tissuemizer Extraction Accelerated Solvent Extractor Soxhlet Extraction Shaker Table Extraction Accelerated Solvent Extractor Soxhlet Extraction Liquid:Liquid Extraction Dilution	EPA 8270D MOD (SIM Mode) GC/MS SOP 5-157
PCBs and Chlorinated Pesticides	Tissue (SOP 5-190) Tissue (SOP 5-307) Tissue (SOP 5-309) Sediment (SOP 5-192) Sediment (SOP 5-307) Sediment (SOP 5-309) Water (SOP 5-200) Products/NAPL (SOP 5-334)	Tissuemizer Extraction Accelerated Solvent Extractor Soxhlet Extraction Shaker Table Extraction Accelerated Solvent Extractor Soxhlet Extraction Liquid:Liquid Extraction Dilution	EPA 8081B MOD and 8082A MOD GC/ECD SOP 5-128
PCB Congeners and Chlorinated Pesticides	Tissue (SOP 5-190) Tissue (SOP 5-307) Tissue (SOP 5-309) Sediment (SOP 5-192) Sediment (SOP 5-307) Sediment (SOP 5-309) Water (SOP 5-200) Products/NAPL (SOP 5-334)	Tissuemizer Extraction Accelerated Solvent Extractor Soxhlet Extraction Shaker Table Extraction Accelerated Solvent Extractor Soxhlet Extraction Liquid:Liquid Extraction Dilution	EPA 8270D MOD (SIM Mode) with elements of Method 1668A and EPA Method 680 for technical details GC/MS SOP 5-315
Alkyl Lead	Products/NAPL (SOP 5-334)	Dilution	EPA 8270D MOD (SIM Mode) GC/MS SOP 5-157
Petroleum Hydrocarbons (range C_8 – C_{40})	Sediment (SOP 5-192) Sediment (SOP 5-309) Tissue (SOP 5-190) Tissue (SOP 5-309) Water (SOP 5-200) Products/NAPL (SOP 5-334)	Shaker Table Extraction Soxhlet Extraction Tissuemizer Extraction Soxhlet Extraction Liquid:Liquid Extraction Dilution	EPA Method 8015 MOD GC/FID SOP5-202
Volatile Organic Hydrocarbons (range C_4 – C_{10})	Sediment and Water (SOP 5-245)	Purge and Trap	EPA Method 8260B MOD EPA 5030B MOD and 5035 MOD GC/MS SOP 5-245

Every day, the people of Battelle apply science and technology to solving what matters most. At major technology centers and national laboratories around the world, Battelle conducts research and development, designs and manufactures products, and delivers critical services for government and commercial customers. Headquartered in Columbus, Ohio since its founding in 1929, Battelle serves the national security, health and life sciences, and energy and environmental industries. For more information, visit www.battelle.org.

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