

# STATEMENT OF QUALIFICATIONS

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**Analysis of Marine and Freshwater  
Sediment, Pore Water, and Tissue Samples**

# ALS Environmental

## Marine Sediment, Tissue, and Water Services Statement of Qualifications

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*Disclaimer: This Statement of Qualifications (SOQ) was prepared to provide our clients with a greater understanding of the sediment and tissue analytical services offered by ALS Services Inc. This SOQ should not be used as a substitute for the regulations and guidance issued by federal, state or local agencies*

## I. Introduction and Overview of Services

ALS Environmental (ALS) provides a full range of analytical services using advanced analytical chemistry procedures to support trace analyses for inorganic and organic constituents in freshwater, seawater, brackish water, sediment, and biological tissue.

A number of ALS laboratories in the U.S. are in their third decade of ongoing maintenance and development of specialized procedures designed to meet the objectives of various projects associated with marine applications. A comprehensive range of sediment programs are supported by the organization, including dredged material projects, contaminated sediment remediation projects, and water quality assessment and compliance initiatives. Typical projects supported by ALS include:

### Dredged Material Management Programs

- Deepening of navigation channels
- Maintenance dredging of dock and harbor areas
- Evaluation of dredged material for beneficial re-use and upland/open water disposal
- Water quality certification and monitoring programs

### Contaminated Sediment Remediation Programs:

- Characterization of impacted sites along river/ harbor/ estuary systems
- Monitoring during impacted sediment remediation construction activities
- Monitoring during post-remedial care/remedy performance evaluations/five-year reviews
- Monitoring to evaluate effectiveness of monitored natural recovery (MNR) programs

### Water Quality Assessment and Compliance Programs:

- Bioaccumulation studies for persistent bioaccumulating toxic compounds
- Ecological and human health risk assessment programs
- Contaminant fate and transport modeling programs
- Total maximum daily load (TMDL) studies
- Fish consumption advisory studies

ALS continues to be a leader in the analysis of sediment, tissue, and water quality samples collected in marine, estuarine, and fresh water environments of port, harbor, bay, river, and estuary systems on a national and international basis. Project environments supported by ALS range from less impacted, coarser-grained sediments of coastal areas, to estuaries, tidal bays, and inland river areas with finer-grained sediments of higher organic content impacted over time by industrial, municipal, and storm sewer sources, marina and boating operations, and atmospheric deposition. Management of sediments in these diverse project environments involves consideration of near-term concerns, such as recreation and navigation, along with potential long-term concerns regarding human health and the environment.

This Statement of Qualifications provides a discussion of the analytical chemistry services provided by ALS. Discussions are provided for sample preparation, extraction, analysis, data reporting, and technical analytical support services provided by ALS. In addition, highlights of relevant project experience, an overview of laboratory facilities, contact information, and quick references for methods/services are provided.

## II. Sediment Management

Sediment management decisions are made within a complex regulatory and technical framework. Projects are subject to often competing and conflicting requirements of federal laws administered by separate federal agencies with additional involvement of state regulatory program requirements. ALS provides scientifically advanced analytical expertise to meet or exceed the complex set of regulatory and technical requirements governing sediment management decisions.

### Regulatory Framework

Sediment management program requirements are defined by several federal agencies that have long recognized the need for a consistent regulatory and technical framework for decision-making. Many sediment management projects are subject to complex, overlapping federal, state, and local requirements. ALS has supported various projects related to regulatory activity by the following organizations:

- U.S. Corps of Engineer (USACE)
- Environmental Protection Agency (USEPA)
- U.S. Fish and Wildlife Service (USFWS)
- National Marine Fisheries Service (NMFS)
- National Oceanographic and Atmospheric Administration (NOAA)
- U.S. Naval Facilities Engineering Command (NAVFAC)
- U.S. Air Force Center for Environmental Excellence (AFCEE)
- U.S. Army Public Health Command (USAPHC)

ALS has become well versed in the performance of sediment program testing in accordance with major federal regulatory requirements, guidance, and protocols, including:

- USACE "Green Book" or "Ocean Testing Manual" - *Evaluation of Dredged Material Proposed for Ocean Disposal Testing Manual (1991)*
- USACE "Inland Testing Manual" - *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. Testing Manual (1998)*
- USACE "Upland Testing Manual" - *Testing Manual for Evaluation of Dredged Material Proposed for Disposal at Island, Near Shore, or Upland Confined Disposal Facilities (CDFs) (2003)*
- USACE *Dredged Material Analysis Tools (2008)*

As federal agencies coordinate with state and regional entities to encourage consistency in decision-making across statutory boundaries with consideration of the technical issues and the continuum of dredged material management options, ALS has gained experience in performing sediment program testing in accordance with several inter-agency protocols. Examples include:

- USEPA Region 10, *Puget Sound Estuary Protocols (PSEP, 1990)* for the Pacific Northwest
- Northwest Regional Sediment Evaluation Team (RSET), *Sediment Evaluation Framework for the Pacific Northwest (2006)*

- USEPA Region 10, USACE Seattle District, and Washington Department of Natural Resources, *Dredged Material Management Program (DMMP)* (formerly *Puget Sound Dredged Disposal Analysis Program or PSDDA*)
- USEPA Region I and USACE New England District, Regional Implementation Manual (RIM) for *Evaluation of Dredged Material Disposal in New England Waters (2004, rev 2010)*
- New Jersey Department of Environmental Protection (NJDEP), Office of Sediment and Dredging Technology, *Dredged Material Management Manual (October 1997; revision in progress)*
- NJDEP *Ecological Screening Criteria for Freshwater Sediments and Surface Water (2009)*

ALS provides region-specific sediment management program expertise in support of dredged material management programs, contaminated sediment remediation programs, and sediment and water quality studies. Our technical skills and expertise assist clients in navigating and managing the various challenges posed by complex and often overlapping sediment program requirements.

## Technical Framework

Dredged material characterizations and sediment cleanup assessments generally involve the use of a variety of physical, chemical, and/or biological information to reach decisions about the presence/absence of risk and how best to manage uncertainty and evident risk when determining appropriate disposal and/or site remediation options. Over the past 25 years, ALS has provided unmatched expertise in the delivery of high quality, scientifically accurate, legally defensible analytical services in support of these programs. Our technical professionals work closely with clients to determine suitable project approaches to meet site-specific project objectives as well as national and regional regulatory requirements for these sediment management programs.

Many sediment management programs require extremely low-level detection limits. This presents unique challenges to the laboratory due to potential analytical interferences related to complex matrices typical of sediment and tissue, as well as marine water. ALS has developed the technical expertise necessary to perform complex ultra-trace analyses in a variety of challenging matrices. To achieve the highest quality results, various analytical processes are incorporated into the methods to assure superior selectivity and sensitivity. These might include unique sample preparation schemes, extractions, and clean-up procedures, as well as specialized instrumental approaches to specifically address a range of sample matrix issues presented by marine and freshwater sediment, tissue, and water.

The following sections of this document provide additional detail related to the various aspects of the analytical chemistry and associated laboratory services typically required to support marine applications. Discussion of sample preparation, extraction, cleanup, instrumental analysis, and related technical services are included.

### III. Preliminary Sample Preparation – Homogenization & Drying

The importance and absolute necessity of thorough homogenization is recognized by ALS and has been an integral part of our Standard Operating Procedures (SOP) for over twenty years of analyzing biological tissue and sediment samples.

**Biological Tissue:** Various types of grinders and blenders are utilized for the homogenization process, depending on the type and size of tissue.

Virtually all non-plant material is subjected to a homogenization step or series of steps prior to freeze-drying in an effort to create a fairly large, representative sub-sample. After freeze-drying, the sub-sample is further homogenized to a fine meal in preparation for removing the final aliquot for acid digestion. Freeze-drying is performed as the preliminary preparation step to subsequent trace metals determinations. Note that, although, ALS occasionally freeze-dries tissues prior to analysis for organics, normal protocol is to use more conventional drying techniques. However, studies have been conducted to demonstrate the technical validity of freeze-drying for certain organic constituents. Also note that moisture is quantitatively determined during the freeze-drying process.

Plant tissue generally undergoes a gross homogenization to obtain a large sub-sample for drying at 60°C as the preliminary step to subsequent trace metals determinations (other than mercury and methyl mercury). The dried material is further homogenized via various techniques depending on the type of tissue. Mechanical milling is normally required. Note that ALS also has the capability to perform cryogenic grinding of plant material, which is the preferred option for preparing samples for determination of mercury and/or organic constituents. Although mercury in plant tissue is generally not volatile at 60°C, aliquots for analysis are taken prior to drying to alleviate doubts. Thus, cryogenic milling of the “as received” plant tissue provides a superior homogenate. Homogenates are air-dried for organics.

**Dissection:** ALS offers biological tissue dissection such as fish filleting, shucking, and specific organ or tissue segregation. After dissection, tissue samples are subjected to homogenization prior to further prep or analysis as previously described.

**Sediment:** Sediment samples are generally mixed in an as received (i.e. wet) state and sub-sampled direct for analysis. Large samples are transferred to suitable containers to facilitate thorough re-mixing of supernatant water when applicable. Drying for organics is done via conventional drying agents. Moisture determinations are performed using standard convection oven methodology.

In some cases trace metals require a true total dissolution that incorporates hydrofluoric acid, involves fusion techniques, or other techniques beyond the scope of typical procedures used most commonly today, calling for various combinations of nitric acid, hydrogen peroxide, and hydrochloric acid. If a true total dissolution is required, then samples are dried at 60°C (or possibly air-dried) prior to being subjected to a more aggressive mechanical milling procedure to reduce particle size sufficient to assure complete dissolution of all material. The milling might be performed via shatterbox (i.e. ring mill), ball mill, cryogenic mill, or mortar and pestle.

**Anaerobic Sample Preparation:** ALS maintains the capability to perform samples manipulations under anaerobic conditions using inert gas purged glove-boxes. This is typically incorporated in the preliminary treatment of sediment when conducting porewater extractions for constituents such as butyltins and others sensitive to aerobic conditions. Another common application is the preparation of samples intended for various sulfide determinations (e.g. acid volatile sulfide – AVS, total sulfide, reactive sulfide, etc.). Loss of sulfide to oxidation during preliminary handling operations should be minimized by working under anaerobic conditions.

## IV. Porewater Extraction, Elutriations, and Sequential Batch Extractions

ALS provides a range of services that includes various extraction protocols that are often used in conjunction with bulk sediment determinations. These procedures generally fall under one of the following:

- Porewater Extraction
- Effluent Elutriate Test (EET) or Standard Elutriate Test (SET)
- Modified Elutriate Test (MET)
- Dredged Elutriate Test (DRET)
- Sequential Batch Extractions
- Selective Sequential Batch Extractions
- Routine EPA Solid Waste Leaching Procedures

### Porewater Extraction

ALS performs traditional pore water extractions using the EPA Region X/U.S. Army Corps of Engineers protocol. Manipulations of sediment samples are performed in a glove box under anaerobic conditions. Double centrifuging is performed in a refrigerated centrifuge, maintaining anaerobic conditions within the sample containers via inert gas blanketing. If filtration is required to support specific project objectives, it's performed using a silver membrane or polycarbonate filter media to prevent loss of butyltin compounds to adsorption.

### Elutriations

ALS supports a variety of elutriations typically applicable to dredging and dredge material management programs conducted in accordance with U.S. Army Corps of Engineers protocols. The procedures include:

- Standard Elutriate Test (SET) / Effluent Elutriate Test (EET)
- Modified Elutriate Test (MET)
- Dredging Elutriate Test (DRET)

### Sequential and Selective Sequential Batch Extractions

ALS supports a range of sequential extractions that are generally customized to address site specific concerns. Although published ASTM methods exist for some procedures, most testing is based on a technical publication or combination of publications. The laboratory is very flexible and versatile in regard to accommodating customize extraction procedures.

In some cases, the procedures simply call for repeated leaching of a sample until equilibrium is reached, while other procedures are selective in regard to targeting specific forms of target analyte. The procedures often target metals, but organics are evaluated for some projects as well.

For metals characterizations, selective extractions might target exchangeable metals, metals combined or bound to carbonates, metals combined or bound to Fe-Mn oxides, metals combined or bound to organic matter, and metals incorporated into the crystalline lattice of silicon based minerals or inert oxides of other metals.

## V. Physical/Geotechnical

ALS supports selected physical and geotechnical test methods associated with sediment management programs. The most commonly required tests are as follows:

- Particle Size Distribution by ASTM D422 (Sieve only, or Sieve/Hydrometer)
- Particle Size Distribution by Puget Sound Estuary Protocol (PSEP)
- Specific Gravity by ATSM C127
- Atterberg Limits by ASTM D4318
- Total Solids by EPA 160.3M
- Total Volatile Solids by EPA Modified 160.4M

Note that Particle Size Distribution tests are often customized to meet certain site specific requirements and/or to be consistent with historic data. Variations to both the ASTM and PSEP procedures are common. ALS routinely customizes the procedure to meet project requirements. This may incorporate using sieve sizes that vary from the published method and/or including more or less size fractions.

## VI. Conventionals

ALS supports selected conventional analytical chemistry test methods associated with sediment management programs. The most commonly required tests are as follows:

- Total Organic Carbon (TOC) by 9060M; ASTM D-4129-82 M; Lloyd Kahn; Plumb; or PSEP
- Speciated Carbon by Laboratory SOP and/or Selected Publications
- Organic Matter by Walkley Black
- Ammonia by EPA 350.1 M; or Plumb
- Total Sulfide by EPA 9030M/SM4500
- Acid Volatile Sulfide (AVS) by EPA Draft 1991
- Acid Volatile Sulfide/Simultaneously Extracted Metals (AVS/SEM) by EPA Draft 1991

**Organic Carbon** characterization is accomplished through various fractionation and measurement techniques that provide partial information on organic matter levels, depending on the selected methodology. ALS supports a full range of analysis of several forms of carbon in water, soil, and sediment including: Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), Fraction of Organic Carbon (FOC), Particulate Organic Carbon (POC), Black Carbon (BC), Total Carbon (inclusive of inorganic carbon), Inorganic Carbon.

**Total Organic Carbon (TOC)** analysis is performed using high temperature dry combustion techniques including SM 5310C (water), EPA 415.1 (water), EPA Method 9060 (water; modify to accommodate solids), EPA Lloyd Kahn, EPA Plumb, PSEP, ASTM D4129-82 (water; modify to accommodate solids), and ASTM D2974 by loss on ignition. ALS also supports the Walkley Black procedure that estimates the amount of organic matter using dichromate oxidation.

**Dissolved Organic Carbon (DOC)** analysis is performed by ALS by several methods including EPA Method 415.1, SM 5310 C, and EPA Method 9060. Determination of DOC is equivalent to TOC, except the sample is filtered prior to preservations and analysis to be selective to dissolved carbon species only.

**Fraction of Organic Carbon (FOC)** analysis is performed using EPA Method 415.1, EPA Method 9060, and ASTM D2974-87. While there is no rigorous definition of the fraction of organic carbon, it is represented as a portion of the organic matter that is available to adsorb the organic contaminants of concern, with less of those compounds available to leach into ground water.

**Black Carbon** analysis is performed the soot quantification method developed by Gustafsson et al. (1997). The Gustafsson et al. method includes inorganic carbon removal, organic matter removal, and dry combustion for carbon quantification. This method involves an acid digestion followed by an 18-hour furnace combustion in the presence of a stream of pure oxygen. ALS has successfully used this procedure for the determination of Black Carbon in freshwater and marine sediments.

**Particulate Organic Carbon (POC)** analysis is performed by ALS on freshwater, estuarine, and coastal waters and marine, brackish, and freshwater sediments using Method 440.0, developed by Zimmerman, et al. (1997). This method includes combustion and then thermal conductivity

detection for elemental analysis to determine particulate carbon (PC) and particulate nitrogen (PN). The method measures the total carbon and nitrogen irrespective of source (inorganic or organic) on the portion of a sediment sample that passes through a number 10 sieve or a 2-mm mesh sieve.

**Total Carbon** analysis is performed on freshwater, estuarine, and coastal waters, as well as marine, brackish, and freshwater sediments using the same methods as TOC, but eliminating the acid treatment that removes inorganic carbon. Note that **Inorganic Carbon** can be determined by eliminating combustion and only measuring evolved carbon dioxide from the acid treatment.

**Ammonia** determinations are performed using published methods that employ one of several options, including ion-specific electrode (ISE) or colorimetric measurement. Pre-treatment of aqueous samples may or may not require distillation to separate ammonia from interfering matrix components. Sediment samples are generally extracted in dilute hydrochloric acid prior to determination by ISE or colorimetric measurement.

**Sulfide** determinations generally target **Total Sulfide** or **Acid Volatile Sulfide (AVS)**. On occasion, **Reactive Sulfide**, **Soluble Sulfide**, and/or variables to other sulfide methods are performed.

Aqueous samples can often be analyzed direct (without preliminary distillation) for sulfide. Most applications associated with marine projects require relatively low detection limits, so the methylene blue colorimetric procedure is preferred. Often the analysis is conducted using a low-level calibration curve and extended cell path to maximize sensitivity. For colored samples, a preliminary distillation (EPA Method 9030) is performed to isolate sulfide from interfering matrix components.

Sediment samples generally require Total Sulfide or AVS. Both procedures incorporate distillations where sulfide is protonated, purged from solution as hydrogen sulfide gas, and then trapped in an alkaline scrubber solution that contains zinc divalent ions to fix the sulfide. The scrubber solution is then treated as per an aqueous sample.

The Total sulfide distillations generally use sulfuric acid, while the AVS procedure uses hydrochloric acid. Both approaches employ relatively high concentrations of acids. Actually, the AVS procedure can be more aggressive due to the ability of high concentrations of chloride to move the solubility equilibrium of metal sulfides to the right by forming metal-chloride complexes, effectively removing reactants from the system. Most often, however, results for AVS and Total Sulfide agree reasonably well.

**AVS-Simultaneously Extractable Metals (AVS-SEM)** determinations are performed by EPA Draft Method 821-R-91-100 and conducted on sediments to provide an indicator of the bioavailability of a number of heavy metals that form metal sulfides with very low solubility product constants ( $K_{sp}$ ). Typically, Cd, Cu, Ni, Pb, and Zn are evaluated, but Ag, Hg, or others are sometimes examined depending on the site. The procedure allows a stoichiometric evaluation of the sulfide versus liberated metals to determine a theoretical estimate of bioavailable metals (i.e. metals present in concentrations greater than the amount of sulfide needed to fix them as insoluble sulfide salts). In addition to performing a standard AVS test,

the residual solution in the distillation flask is quantitatively analyzed for heavy metals of interest. For convenience, metals and sulfide are reported in units of chemical equivalents.

## VII. Trace Metals – Digestions/Chemical Separations/Instrumental

ALS performs analysis for metals and metalloids utilizing numerous procedures and/or variations of procedures to meet the objectives of the project. Often, ultra-trace concentrations of particularly toxic elements must be determined, so specialized preparation and analysis techniques are employed. Metals determinations are typically performed by following published methods, but refined processes are incorporated into the procedures within the scope of the methods to maximize sensitivity and selectivity.

Careful and thorough digestion of samples for trace metals is critical to obtaining accurate results. For many applications, ultra-trace determinations of micronutrients and/or highly toxic elements are required. Minimizing contamination during the digestion process is critical. In addition, potential complications from incomplete carbon removal can impact the results. For determination of ultra-trace levels of metals in high saline samples, chemical separations are generally required. Although pre-quadrupole chemistry performed in conjunction with some instrumentation circumvents many interferences, care must be taken to avoid over-taxing that technique.

Following sample digestion, instrumental analysis is performed for trace metals constituents using Inductively Coupled Plasma–Mass Spectrometry (ICP–MS), Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP–OES), Graphite Furnace Atomic Absorption (GFAAS), Gaseous Hydride Atomic Absorption (GHAAS), Flame Atomic Absorption Spectroscopy (FAAS), Cold Vapor Atomic Absorption (CVAAS), and/or Purge & Trap Cold Vapor Atomic Fluorescence Spectroscopy (P&T–CVAFS). For most applications ICP–MS and/or ICP–OES are sufficient for all elements except Hg. For Hg, either CVAAS or P&T–CVAFS are adequate, depending on the detection limit requirements. These instrumental methods are used for water, sediment, and tissue.

**Aqueous Digestion** is performed using conventional methods, unless more complexity is mandated by the project plan. Freshwater samples are digested using procedures incorporated in the instrumental procedures or by EPA Methods 3005 or 3010.

Decisions on which digestion procedure are generally made at the laboratory based on visual inspection of the samples. If significant solid material (particulate, sediment, biota or partially degraded biota, etc.) is observed, the more aggressive of the procedures is applied to assure total oxidation of organic material and/or concentrated acid treatment of particulate. If the sample is relatively free of solid material and color, a soft digestion is performed.

Note that the standard digestions do not assure complete dissolution of certain types of particulate (silicon based minerals, selected metal oxides, etc.) without treatment with hydrofluoric acid. If a digestion procedure that incorporates hydrofluoric acid (HF) is required, ALS performs several variations of procedures that utilize HF.

If samples are high in dissolved salts, either dilutions or chemical separations are mandated. The level of salts might be low enough to simply dilute without jeopardizing project objectives relative to detection limits. If dilution is not feasible, several options using chemical

separations to remove interfering salts are available. Further discussion of addressing physical and chemical interferences is included in later sections of this discussion.

**Sediment Digestion** is most often done using conventional procedures. Assuming a procedure that utilizes nitric acid, hydrogen peroxide, and hydrochloric acid is adequate to meet the objectives of the project; the samples are typically digested using EPA Method 3050. The procedure is normally followed as per the method.

If a total dissolution is required, EPA Method 3052M is most often used depending on detection limit requirements. For ultra-trace applications, an in-house procedure is used to obtain lower detection limits. That procedure equates to a chemical separation where total dissolution is accomplished via treatment with excess hydrofluoric acid. Silicon is removed as volatile  $\text{SiF}_4$  along with residual hydrofluoric acid. The residue is reconstituted with nitric acid to produce a relatively clean solution appropriate for essentially any instrumental technique. In addition to the HF procedures, fusions are also performed at ALS Environmental, most commonly a lithium borate procedure.

**Tissue Digestion** is generally handled consistently the same. Once final milling of the freeze-dried homogenate is complete, the sample is ready for sub-sampling for digestion. Digestions are typically performed using ALS SOP, which consists of a nitric acid procedure conducted in closed Teflon vessels at 105°C. The heating is done in convection ovens over a period of approximately 16 hours. The extended digestion time allows complete oxidation of organic material so dissolved carbon levels are minimized, which reduces or eliminates several carbon induced interferences for both ICP/OES and ICP/MS determinations.

**Clean Room Preparation** at ALS is performed in a Class 1000 clean room equipped with Class 100 laminar flow fume hoods for low-level and trace level metals digestions, as well as preparation of field sampling supplies. The positive-pressure facility was specially designed and built with metals-free countertops, flooring, walls, hoods, and ductwork to support element-free sample preparation for ultra-trace metals determinations.

**Chemical Separations** are performed using various techniques depending on the scope of the project. Occasionally, sediment samples are subjected to chemical separations as previously mentioned, as is true for tissues. The most common chemical separation for sediment and tissue is the use of hydride generating procedures. Although not used extensively, some applications still require it. In addition, maintaining capability to perform hydride procedures gives the laboratory the ability to confirm questionable detections by a second or third procedure.

The most common use of chemical separations is for the analysis of seawater or other high saline aqueous samples. ALS maintains several procedures for the analysis of seawater, but the most universal methods with the best overall performance for a relatively wide range of elements are the reductive precipitation technique and the on-line chelation technique. Both procedures are listed in EPA Method 1640.

The on-line chelation procedure is often sufficient for many projects. The procedure employs a chelating resin in line with the aqueous sample flow. Metals of interest that follow the chelation mechanism are retained by the column while major matrix components are either

sent to waste, or diluted and sent to the plasma for determination of other elements. The chelated metals are eluted off the column with dilute nitric acid and directed to the plasma for analysis. The analysis of elements determined via dilution is aided by pre-quadrupole chemistry using the latest Perkin Elmer Dynamic Reaction Cell (DRC) technology. The lowest limits are realized for the chelated elements, but others are generally sufficient to meet most project specifications.

The reductive precipitation procedure incorporates a chemical separation to remove interfering matrix components so final analysis of metals can be performed using ICP-MS. The separation utilizes reduction of certain target analytes to the elemental state and precipitation of others as the boride depending on reduction potentials and/or boride solubility. The precipitation is facilitated using elemental palladium and iron boride as carriers. Once separated from the seawater matrix via centrifugation, the precipitate is re-dissolved and analyzed using ICP-MS.

ALS has effectively used these sample preparation techniques to support ultra-low level determination of metals in seawater and other saline samples to meet risk-based project objectives and permit-based regulatory compliance criteria.

**Instrumental Procedures** are generally limited to EPA Methods 200.8/6020/1638 for ICP-MS, 200.7/6010 for ICP-OES, 7470/7471 for CVAAS, and 1631 for P&T-CVAFS. EPA Method 7742 is often the method of choice for Se when required. In situations where GFAAS and/or FAAS are used, SW846 7000 series methods are used.

Instrumental procedures are capable of producing incorrect data that can be virtually impossible to identify when evaluated via third party validation or other mechanism. The associated QA/QC measurements add value and certainly provide information related to the data, but they do not necessarily identify issues associated with sample matrix. A matrix spike recovery in an acceptable range does not necessarily indicate an absence of interference, particularly from a high bias standpoint.

In addition to the discussions in the EPA procedures concerning interferences and the prescribed means of monitoring and/or performing corrections, ALS generally incorporates an additional level of technical quality assurance. The multi-element ICP procedures in particular are capable of producing erroneous results, despite following the minimum requirements cited in the associated methods. Thus, additional care is taken to minimize the potential for interferences to go unnoticed.

Complete digestion of samples is important from the standpoint of assuring dissolution of all elements, but even with complete dissolution, certain elements are adversely impacted by incomplete oxidation of organic material. As previously mentioned, incomplete removal of dissolved carbon will result in enhanced ionization of Se and As in the plasma, which impacts both ICP/MS and ICP/OES. Correction for either technique is essentially impossible, so the most practical means of minimizing this interference is complete oxidation of organic material. Carbon also combines to form various polyatomic ions that result in isobaric overlaps on the ICP/MS, so minimizing its presence improves accuracy.

For ICP/OES, common spectral overlaps are addressed using inter-element factors derived from experimental data. Care is taken to assure the factors are derived from actual spectral overlap rather than impurities in the salt or purchased standard solution used for determining the interference. In addition to the commonly controlled spectral interferences, additional factors are used because multiple emission lines are monitored for many elements. Note that ALS routinely runs a second correction on a real-time basis when significant interference is observed (i.e. the magnitude of the correction is large relative to the signal from the actual analyte). This essentially equates to a fine-tuning of the correction by measuring the impact of the particular interfering element, generating a second factor, and manually applying the correction to the observed value at the time of analysis. Inter-element correction factors are preserved over time by assuring ion:atom ratios remain constant from day to day, but even at that, the factors are not perfect.

Primary analytical emission lines are generally used for quantifying results, but secondary or tertiary lines are monitored for comparison reasons. This allows identification of physical and/or chemical interferences that impact one line, but not another. Careful inspection of the complete optical emission fingerprint of each sample is important to assuring quality results. Although line switching is generally done to extend linear range, occasionally the reason is interference.

Evaluating physical interferences associated with ICP/OES determinations is generally done via internal standardization (added post-digestion). Certain elements are impacted more severely by dissolved salts during droplet formation and subsequent uptake to the plasma. However, the impact is generally well characterized for sediment and tissue. Aqueous samples impacted by seawater intrusion or other sources of salts represent situations where monitoring of internal standard response is of most value. Normally, a reduction in signal is observed, so post-spike evaluation yields more specific information across the range of elements measured.

For ICP/MS determinations, common interference equations reside in the operating system to correct for known isobaric interferences. These are generally effective, but not all are equally effective. For example, low concentration chromium in biological tissue should not be analyzed using ICP/MS (without collision or reaction cell technology) due to argon-carbon overlap, as well as imperfect chloride-related interference corrections. Despite efforts to minimize carbon in the digestates, enough remains to impact low concentrations of chromium (i.e. elevated recovery of Cr in the NRC TORT-2 and DORM-3 tissue SRMs). The other significant impact of carbon is on arsenic. A mechanism in the plasma has been shown to enhance ionization of arsenic, which results in a high bias. This has been substantiated at ALS and corrected via carbon removal as discussed. Other uncorrectable interferences exist for several other elements, so switching to either ICP/OES or ICP/MS with collision cell or reaction cell technology is required. A couple examples include the determination of vanadium in the presence of elevated chloride and cadmium in the presence of high molybdenum concentrations. Of course many other examples exist with varying degrees of impact on the data. The laboratory spends considerable effort assuring interferences are corrected where possible, but also significant effort assuring uncorrected interferences are identified during analysis and/or review.

Using a similar mindset as described for ICP/OES where an optical emission fingerprint is generated, multiple isotopes are monitored for every element where practical for ICP/MS.

Exceptions are elements with only one naturally occurring isotope or minor isotopes of little to no analytical value. However, many elements have multiple usable isotopes that provide important information relative to recognizing interferences. Some of the mid-mass elements are particularly important. One of the keys to recognizing interference is to monitor multiple elements (target and non-target) and isotopes, so a comprehensive fingerprint of each sample is generated.

**Total Mercury** is performed at ALS by EPA Methods 7470 and 7471 or EPA Method 1631 in freshwater, seawater, brackish water, marine sediment, and tissue samples. The choice of methods is dictated by the required detection limit for the project.

**Metals Speciation** incorporates many of the procedures described for total metals, although some procedures vary radically, while others simply include a preliminary chemical separation to isolate the specie of interest.

**Methylmercury** is performed using EPA Method 1630 in marine, brackish, freshwater, and porewater samples by distillation, aqueous ethylation, purge and trap, chromatographic fractionation, and CVAFS. The procedure is modified to accommodate sediment and tissue by incorporating a preliminary solvent extraction prior to ethylation and subsequent analytical processing. Tissues receive an extraction with alcoholic KOH and sediments are extracted with methylene chloride. ALS has successfully implemented special sample handling procedures in order to achieve ultra-trace level method reporting limits for Methylmercury in waters, tissues and sediments.

**Arsenic Speciation** at ALS supports analysis for As(III), As(V), MMAs, and DMAs by EPA Method 1632 by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry. Alternatively, LC/ICP/MS with reaction cell technology by ALS SOP can be used for arsenic speciation.

**Selenium Speciation** is performed for Se(IV) and Se(VI) using LC/ICP/MS with reaction cell technology by ALS SOP.

**Chromium** speciation for Cr(VI) and Cr(III) is performed in sediments, freshwater, seawater, and porewater by EPA Method 7196 using colorimetric determination with discrete-FIA detection, as well as by EPA Method 7199 using ion chromatography. Additionally, Cr(VI) is performed using EPA Method 7195/7191, which incorporates a chemical separation using a co-precipitation technique followed by GFAAS analysis.

ALS also supports low-level analysis of Hexavalent Chromium in water by Ion Chromatography techniques using EPA Method 218.6 and EPA Method 218.6 (Low Level).

**Butyltins (mono, di, and tri)** are determined in freshwater, seawater, porewater, sediment, and tissue. Samples are analyzed using solvent extraction, derivatization, extract cleanup by elution through alumina and silica gel columns, and instrumental analysis by Gas Chromatography Flame Photometric Detector (GC/FPD) with secondary column confirmation by GC/FPD or GC/MS. The procedures are consistent with C.A.Krone, et al. and M.O. Stallard, et al. ALS synthesizes the Grignard Reagent used in this procedure, as commercially available reagents offer unacceptable purity. A minimum of ten percent of analyte detections are confirmed by secondary column GC/FPD or GC/MS analysis, with all detectable values

confirmed for samples originating from an uncharacterized site with no historical data to suggest the likelihood of the presence of butyltins.

## VIII. Organics – Extraction/Cleanup/Instrumental

ALS offers a wide range of organic sample extraction, cleanup, and instrumental options to support sediment, tissue, and water quality programs. The services are designed to support a range of sediment management program objectives, with the primary requirement of the analytical chemistry laboratory to provide methodology with appropriate sensitivity and selectivity.

Often, ultra-trace concentrations of particularly toxic compounds must be determined, so specialized preparation and analysis techniques are employed. Organic determinations are typically performed by following published methods, but refined processes are incorporated into the procedures within the scope of the methods to maximize sensitivity and selectivity. In addition, ALS offers a number of procedures outside the scope of published methods. In these cases, in-house SOPs are developed. Note that ALS holds certifications for a number of laboratory specific SOPs.

Careful and thorough extraction of samples for trace organic constituents is critical to obtaining accurate results. For many applications, ultra-trace determinations of highly toxic compounds are required. Minimizing contamination during the extraction and cleanup process is critical. For determination of ultra-trace levels of organics in complex sediment and tissue matrices, effective cleanups must be employed to maximize selectivity during the instrumental determinations.

Following sample extraction and cleanup, instrumental analysis is performed for organic constituents using a combination of Gas Chromatography/Mass Spectrometry (GC/MS), GC/MS with Selected Ion Monitoring (GC/MS/SIM), GC/MS with Large Volume Injection (GC/MS/LVI), GC with tandem mass spectrometry (GC/MS/MS), GC with High Resolution Mass Spectrometry (GC/HRMS), GC with Electron Capture Detection (GC/ECD), GC/ECD with LVI, GC with Flame Photometric Detection (GC/FPD), GC with Flame Ionization Detection (GC/FID), Liquid Chromatography with Ultraviolet Detection (LC/UV), and LC with tandem mass spectrometry (LC/MS/MS).

A comprehensive chemistry package for many projects includes a combination of a number of procedures, depending on the detection limit requirements and compound lists. These instrumental methods are used for water, sediment, and tissue.

**Organic Extractions** performed at ALS generally fall under one or more of the following techniques, depending on the project specifications:

- Soxhlet Extraction (Manual & Automated)
- Sonication Extraction
- Pressurized Liquid Extraction (PLE)
- Solid Phase Extraction (SPE)
- Continuous Liquid-Liquid Extraction
- Separatory Funnel Extraction
- Solid Phase Micro Extraction (SPME)

Automated Soxhlet or manual Soxhlet extractions are performed for virtually all sediment and tissue samples. Segregated systems for low level work are employed to assure minimal background contamination of target and non-target components. Of particular note is the added precautions when preparing samples for GC/ECD analysis (e.g. low level pesticides, PCBs, etc.). Minimizing background components is critical to maximizing selectivity. Ultra-trace applications call for normal decontamination procedures, plus baking of glassware.

Continuous liquid/liquid and SPE are used for most aqueous applications, although separatory funnel extractions are also performed. Certifications are maintained for all procedures to allow flexibility to tailor the needs of the project to fit the appropriate analytical technique.

Extractions procedures at ALS generally fall under SW846 methodologies, but refined processes are incorporated into the procedures within the scope of the methods to maximize sensitivity and selectivity. A limited number of procedures fall under ALS SOP, but certifications are generally in place for in-house methods.

In addition to conventional solvent extractions, EPA Method 8272 is performed. The SPME portion of the process is included in the method. The procedure is intended for direct extraction of PAHs onto a SPME fiber that is subsequently loaded on a thermal desorption sample introduction system interfaced to a GC/MS system. Once on column, PAHs are chromatographed, detected, and quantified as per other GC/MS/SIM determinations.

**Extract Cleanup** is always performed for sediment and tissue. Aqueous samples are considered for cleanup on a case to case basis. Extensive cleanup of sample extracts is necessitated by various factors, such as:

- Biological macromolecules (e.g., residue from algae, fish, crustaceans, mammals, etc.)
- Naturally occurring lipids in fish tissue
- Partially hydrolyzed biogenic material from various natural and non-natural sources
- Sulfur from anaerobic microbial reduction of sulfate in sediment
- Petroleum related contamination in sediment

Sample extract cleanup protocols supported by ALS include:

- Gel Permeation Chromatography (GPC) Cleanup
- Silica Gel Cleanup
- Alumina Cleanup
- Florisil® Cleanup
- Carbon Cleanup
- Permanganate Cleanup
- Mercury or Copper Cleanup
- Acid Cleanup

ALS always applies sample extract cleanup techniques for sediment and tissue prior to instrumental analysis of Semi-volatile Organic Compounds (SVOC), Polyaromatic Hydrocarbons

(PAH), Organochlorine Pesticides, and Polychlorinated Biphenyls (PCB). Extracts receive GPC initially, prior to secondary and tertiary treatments. The exception is PAHs, where GPC is bypassed in favor going straight to silica gel fractionation. Multiple cleanup steps are common when attempting to achieve low level project reporting limits. The selected cleanups depend on the analytes of interest, anticipated sample matrix effects, and project criteria. The objective is to maximize the clarity of chromatographic separations by reducing background contribution. This works towards maximizing selectivity and sensitivity to produce reliable results for ultra-low reporting limits typical of sediment and tissue programs.

Secondary and tertiary cleanups commonly employed include Florisil for pesticides and PCBs, sulfuric acid for PCBs and selected pesticides (inert to the treatment), mercury for removal of elemental sulfur for pesticides and PCBs, carbon for additional removal of non-target organic material (pesticides), and alumina (pesticides) as an option when Florisil® is ineffective.

Silica gel is used to separate analytes from interfering compounds of a different chemical polarity. Fractions are collected that represent varying affinities to the column material. This cleanup is used extensively for PAHs, but also for derivatized phenols, and separation of organochlorine pesticides and PCBs.

Alumina is a highly porous and granular form of aluminum oxide. Alumina is used at varying pH ranges (basic, neutral, and acidic) for cleanup of sample extracts.

Permanganate/sulfuric acid cleanup is a rigorous preparation used typically on extracts prior to PCB analysis. The preparation removes various pesticides that may interfere with PCB analysis, as well as other organic material that causes a detector response.

Elemental sulfur is encountered in various extracts and will interfere with the PCB and pesticide analysis. The treatment of the extract with elemental mercury or un-oxidized copper powder will usually remove the sulfur interference. Multiple treatments are not uncommon.

For GC/HRMS procedures (PCB Congeners and Dioxin/Furan), cleanups are incorporated into EPA Methods 8290, 1613, and 1668. The ALS GC/HRMS laboratories conform to the methods.

**SVOCs/PAHs/Alkylated PAHs/Biomarkers** are all typically analyzed using a variation of EPA Method 8270, a GC/MS procedure applicable to a wide range of SVOCs. Note that incorporating SIM and LVI into the method falls under the allowances of SW846 as long as performance is demonstrated. For low-level semi-volatile determinations that include a range of chemical classes (e.g. phenols, phthalates, PAHs, etc.), conventional full-scan GC/MS techniques are used in conjunction with LVI. Preliminary cleanup is generally restricted to GPC by the range of functional groups represented in a comprehensive scan. However, GPC is effective enough to allow the LVI to put a greater quantity of extract (i.e. analyte) on column to improve detection limits.

ALS specializes in PAH analysis with trace-level analytical capability in water, sediment, and tissue matrices. This includes 'extended' compound lists of alkylated daughter PAHs. Clean laboratory techniques are used to minimize background contamination for trace analytical concentrations and thorough cleanup techniques are employed to reduce matrix effects of interfering analytes. False positives and/or elevated detection limits are essentially eliminated via the correct use of specialized cleanup procedures. Alkylated PAH analyses are often used

in identification of source contamination. ALS has been performing alkylated PAH determinations since the mid-1990's, and has continuously expanded the target analyte list to meet emerging needs.

In addition to PAH and alkylated PAH determinations, ALS provides a relatively extensive list of biomarkers typically used for forensic investigations.

**PAHs in Porewater by SPME/GC/MS/SIM** is offered in addition to conventional solvent extractions and conventional analysis techniques. The determination follows EPA Method 8272 or ASTM Method D7363-07. The SPME portion of the process is included in the method. The procedure is intended for direct extraction of PAHs onto a SPME fiber that is subsequently loaded on a thermal desorption sample introduction system interfaced to a GC/MS system. Once on column, PAHs are chromatographed, detected, and quantified as per other GC/MS/SIM determinations.

**Pesticides/PCB Aroclors/PCB Congeners (by GC/ECD)** are determined in sediments, tissue, seawater, and porewater by EPA Method 8081(Pesticides) and 8082 (PCB Aroclors and Congeners), which are both GC/ECD procedures. To achieve ultra-low level reporting limits for Pesticides and PCBs by GC/ECD, adjustments within the allowance of SW846 are made to sample mass, final volume, range of calibration curve, and injection volume. In addition, extensive cleanups are performed as appropriate to minimize background components that cause the ECD to respond. An LVI is used to deliver additional extract volume for low level PCB congener analysis.

For PCB Aroclors, a list of nine Aroclor mixtures is available. For PCB congeners, report lists include the NOAA list of PCB congeners, National Institute of Standards and Technology (NIST) list of 18 PCB congeners, USACE list of PCB congeners, and the World Health Organization (WHO) list of 64 PCB congeners.

**Pesticides/Low Level (by GC/MS/MS)** are analyzed as an option to the conventional approach that employs GC/ECD. ALS has procedures for both Organochlorine and Organophosphorus Pesticides by GC/MS/MS. For Organochlorine Pesticides, an isotope dilution procedure is used that mimics EPA Method 1699. The only significant difference is the use of GC/MS/MS instead of GC/HRMS. Although the GC/MS/MS does not have the sensitivity of HRMS, the improvement in selectivity over GC/ECD techniques is glaring. In addition, sensitivity is also improved. The end result is the ability to reliably determine trace levels of pesticides at low concentrations in complex matrices. With the second quadrapole, uncorrectable co-elutions on an ECD are eliminated.

Use of 1699M eliminates PCB congener interference, which can cause false positive identification and/or over estimation of analyte concentrations for pesticides. The triple-quad LVI/GC/MS/MS approach provides excellent selectivity in complex matrices by generating unique spectral fingerprints that result in an unambiguous identification resulting in increased data defensibility. This enhancement further reduces co-extractable interferences. In addition to the selectivity and sensitivity gains, use of isotope dilution improves accuracy of final results.

For the Organophosphorus Pesticides, EPA Method 8270 is cited. Again, the ability to sort through complex background matrix components via the second quadrupole dramatically improved selectivity, as well as sensitivity.

**PCBs Congeners/Homologs (by GC/HRMS)** are analyzed for the full list of 209 congeners by isotope dilution with GC/HRMS in freshwater, seawater, brackish water, sediment, and tissue. ALS implements extensive sample handling procedures designed to minimize contamination in order to achieve ultra-low detection limits. In addition to the cleanups described in the method, alumina treatment is performed on a case by case basis as needed. to minimize matrix interferences for low level detection of PCB congeners in sediment and tissue sample analysis.

ALS reports PCB congener data along with Toxicity Equivalency Factors (TEF) that estimate the toxicity of dioxin-like PCBs to that of 2,3,7,8-Tetrachlorodibenzodioxin in accordance with USEPA procedures. Detection limits for GC/HRMS applications are derived from signal to noise calculations for each analyte on an individual sample basis. Thus, an MDL can be reported, but it has little to no relevance to this application. The calculated detection limits are reported with each sample result and are generally much lower than the associated method reporting limits (calculated from the low point in the calibration curve).

The full list of 209 congeners can be reported, or a subset of congeners, depending on the project requirements. Homologs can be reported instead of congeners or in addition to congeners. EPA Method 1668 is cited for all matrices.

**Dioxins/Furans (by GC/HRMS)** are analyzed by isotope dilution with GC/HRMS in freshwater, seawater, brackish water, sediment, and tissue. ALS performs dioxin and furan analysis by EPA Method 8290 or 1613. The laboratory has developed extensive sample handling procedures designed to minimize contamination in order to achieve ultra-low detection limits. The procedures minimize sample transfer and rely on disposable lab ware where feasible. The cleanup procedures defined in the methods are followed. Additional clean-ups are employed on a case by case basis as needed for exceptionally complex matrices.

ALS reports sample analytical data along with calculation of the 2,3,7,8-TCDD Toxicity Equivalency Factor (TEF) in accordance with USEPA procedures. Detection limits for HRGC/HRMS instruments are calculated for each congener in each sample during each analytical run. Thus, an MDL can be reported, but has little to no relevance to this application. The calculated detection limits are reported with each sample result and are generally much lower than the associated method reporting limits (calculated from the low point in the calibration curve).

### **Other Organic Contaminants of Concern**

**Pharmaceutical and Personal Care Products (PPCPs):** ALS provides a full range of analytical services for residual compounds originating from PPCPs. These compounds can be detected at ultra-low concentrations using EPA Method 1694 and modern LC/MS/MS instrumentation. Many of the compounds analyzed have negative effects on the endocrine systems of aquatic organisms, humans, and wildlife. Due to their chemistry, many are not amenable to GC and GC/MS techniques. In 2004, ALS initiated the development of procedures for analyzing PPCPs

using isotope dilution with solid phase extraction and LC/MS/MS determination. The procedures have been refined and modified to meet EPA Method 1694 requirements when it was released some time after ALS completed initial method development. The list of available compounds has continued to evolve over the years and still remains in a state of flux as new compounds are regularly added.

**Perfluorinated Compounds (PFOA, PFOS, etc.):** ALS performs analysis of perfluorinated compounds using solid phase extraction and LC/MS/MS. The most notable compounds are Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS), although a full list of compounds from penta thru octa are determined. The compounds belong to a family of perfluoroalkyl acids (PFAA) that have a fluorine substituted carbon and a sulfonic or carboxylic acid functional group. These compounds have historically been used in fire fighting foam, fabric & carpet stain repellent, semiconductor industry materials, paper coating, fast food wrappers, lubricants, flame resistant hydraulic fluid, and plastics. The compounds are considered persistent and bioaccumulative. The procedures employed by ALS produce superior sensitivity and selectivity.

**Brominated Flame Retardants (PBDEs & PBBs):** ALS provides analytical services for a class of compounds referred to as brominated flame retardants (BFRs), which includes Polybrominated Diphenyl Ethers (PBDE) and Polybrominated Biphenyls (PBB). These compounds have been used globally as flame retardants for several decades in a variety of consumer products such as electrical appliances, wires, cables, building materials, resins, laminates, textiles, toys, cars, planes, etc. They have gained increased attention because of their persistence in the environment, potential for bioaccumulation, and potential toxicity. ALS developed special analytical procedure for the determination of PBDEs and PBBs based on continuous liquid-liquid extraction or solid phase extraction with silica gel and acid cleanups, and determination by EPA Method 8270 using GC/MS/SIM/LVI techniques to maximize sensitivity.

In addition to the standard GC/MS/SIM/LVI procedure, ALS performs PBDE determinations using GC/HRMS according to EPA Method 1614. The GC/HRMS procedure provides superior selectivity and sensitivity. As with other GC/HRMS procedures, Method 1614 uses isotope dilution to improve accuracy and precision.

## IX. Detection Limits

Tables of detection limits are readily available on request (refer to “Contacts” section). Method Detection Limit (MDL) studies are performed as per 40 CFR Part 136, Appendix B and updated on a regular basis consistent with regulatory requirements.

The procedures described in this document are geared toward obtaining trace to ultra-trace limits in difficult matrices with the overall intention of meeting various regulatory requirements associated with specific applications. The need for improved selectivity and sensitivity relative to meeting continuously evolving regulatory criteria is at the forefront of ALS services.

Tables of detection limits are available in electronic form as an Excel file. The file is in a relatively constant state of flux as new limits are established, new procedures come on line, and upgrades to current systems and processes impact detection limits. In addition, numerous options within a single analytical method are available that allow customized matching of the appropriate method variation to the specific application.

In general, ALS consistently develops methods and/or improves existing methods to meet virtually any detection limit requirement requested. Occasionally, an exception occurs, but the general rule is ALS can meet regulatory limits.

## X. Data Management and Reporting Services

ALS has successfully met the data management requirements of a wide variety of projects for both government and private sector clients. Included in this section is a description of our information system platform and electronic data deliverables. ALS laboratory facilities operate in a server/local area network (LAN) configuration. The organization has standardized on Microsoft (MS) Window applications (i.e., Outlook, Word, Excel, Access, and MS Exchange).

### Laboratory Information Management System (LIMS)

ALS facilities are implementing a single laboratory information management system (LIMS) that utilizes an Oracle® database environment to track samples from time of receipt to data reporting to allow for sample and data tracking across the network. Samples are logged into the LIMS from the information provided on the Chain of Custody, including holding time information, project turnaround time, required QA/QC, report due dates, etc. Client-specific information can be accessed during login and correlated to specific projects for that client. Information for the project is communicated to the appropriate laboratory personnel electronically and provides an initial set of information that will be linked to the analysis and reporting. As each laboratory completes the analyses of samples on any project, the information resides in LIMS. Daily reports are generated for the individual department within the laboratory to keep appropriate personnel apprised of the status of samples and projects in-house. The LIMS has the capability of monitoring multiple due dates within a project (combinations of standard / rush turnaround times within the same sample delivery group).

### Data Acquisition and Reporting

Analytical instruments at ALS produce data files that are imported into various data reduction and data validation tools. The master file is accessed by a proprietary program called Electronic Data Deliverable Generator (EDDGE). The EDDGE program normalizes the data and translates applies the pertinent revisions to valid values, fields, formats, etc.

All ALS laboratory data is acquired directly to a local area network or locally and then transferred to a centralized acquisition server from which all reporting takes place. There are a few tests that require manual entering of data because the test itself cannot be automated. Once all data is gathered, the hardcopy report is generated along with an electronic data deliverable (EDD). The EDD and report reports are generated from the same data source.

ALS offers a full range of EDDs generated in various commercial formats (e.g., EQuIS, GIS-KEY, SEDD, etc.), client-specific formats, and Federal Programs (DOD Army, Navy, and Air Force) deliverable formats to support data transfer into large relational database management systems. ALS works closely with clients to produce electronic data formats compatible to conform to strict data structures and fields. Over 500 different EDD formats are supported as of 2012 and the number continues to grow. A team of data management specialists produce

both EDDs and hard-copy reports. In addition, a development team of IT specialists performs updates and creates new formats as requested.

## XI. Regional Project Experience

ALS has routinely performed chemical analyses in support of major national and regional regulatory protocols, including those issued by: USACE, USEPA, Pacific Northwest / Puget Sound, Los Angeles / Southern California Ports and Harbors, Boston Harbor, Great Lakes Region, Lower Passaic River / Raritan Bay New Jersey, Gowanus Canal / New York Harbor and Water Ways, Lake Onondaga / Upstate New York, Hudson River New York, Deep Water Horizon / Gulf of Mexico, Florida Everglades, Louisiana Bayous, North/South Carolinas, Chesapeake Bay, etc. A brief summary of ALS project experience dating back to several decades of sediment management program services is provided in the sections below.

### Pacific Northwest / West Coast Region

Over the past 25 years, ALS has provided sediment program analytical services for projects conducted in the Pacific Northwest / West Coast Region. These services were conducted in accordance with national, regional, and/or local regulatory and technical protocols issued by: USACE, USEPA, Puget Sound Estuary Program (PSEP), Puget Sound Dredged Disposal Analyses (PSDDA), Puget Sound Water Quality Authority, State Departments of Ecology, California EPA, and Regional Port and Harbor Authorities. These projects have included analyses of sediment, tissue, and water samples for a variety of trace metals, organics, geophysical, and conventional constituents. Highlights of ALS regional sediment program experience includes:

- Midway Atolls, Hawaii / Sediment Investigation
- Pearl Harbor, Hawaii / US Navy /West Loch Dredge Sediment, Porewater, Elutriate Study
- Alaska River Bioaccumulation Study / Alaska Department of Fish and Game
- Ketchikan, Alaska Pulp Company / Totem Marine Sediment Investigation
- Columbia River Estuary Study, Portland Oregon
- Ross Island Sand and Gravel Company, Portland, Oregon / Remedial Investigation
- Port of Portland, Oregon / Sediment / Tissue / Marine Water Study
- Port of Newport, Oregon / Sediment Dredge Material Characterization
- Puget Sound, Washington Confined Disposal Site Study
- Puget Sound, Washington / NOAA BioEffects and Status and Trends Studies
- Rayonier Pulp Mill and Landfill, Port Angeles, Washington / Remedial Investigation Program
- Duwamish River, Seattle Washington / Sediment Characterization / Water Quality Assessment
- Hylebos Waterway Cleanup Committee Investigations, Commencement Bay / Near Shore / Tidal Flats Superfund Site, Southern Puget Sound, Tacoma, Washington
- Port of Kalama, Washington / Sediment and Pore Water Investigations
- Tongue Point, Astoria, Oregon / Naval Air Station Finger Pier / Sediments Investigation
- McCormick and Baxter Creosoting Company, Stockton California / Remedial Investigation
- Hugo Neu-Proler, Wilmington California / Sediment Investigation
- Port of San Diego, Chula Vista Dredge, California / Sediment / Marine Water Study
- San Francisco, California / US Army Corps of Engineers / Sediment Monitoring Program
- Port of Los Angeles, California / Operable Units 2 and 3

- San Francisco/ Oakland Bays, California / Ship Dry Dock Sediment/ Water/ Wipe Investigation
- San Francisco Bay, California / NOAA BioEffects and Status and Trends Studies
- San Diego and Southern California Bays / NOAA BioEffects and Status and Trends Studies

## Mid-West / Western / Great Lakes Region

ALS has provided sediment program analytical services for projects conducted in the Midwest / Western / Great Lakes Region. These services were conducted in accordance with national, regional, and/or local regulatory and technical protocols, including: USACE; USEPA; State Departments of Environmental Quality and EPAs; Great Lakes Legacy Act of 2002; Great Lakes Water Quality Agreement (USA and Canada); International Joint Commission of Canada and USA. These projects have included analyses of sediment, tissue and water samples for a variety of trace metals, organics, and conventional chemical constituents. Highlights of ALS regional sediment program experience includes:

- Grand Calumet River PRP Group / Gary, Indiana; Chicago, Illinois; Lake Michigan
- Manistique Harbor and River, Michigan / Sediment Remediation and Monitoring Program
- Oil Pipeline Release Response, Billings Montana / Sediment Remediation and Monitoring
- Cuyahoga River, Ohio / Sediment Remediation and Monitoring Program
- Ashtabula River, Ohio / Sediment Remediation and Monitoring Program
- Duck and Otter Creek Toledo, Ohio / Dredge Material Management Program
- Camp Perry Military Base and Ottawa National Wildlife Refuge, Port Clinton, Ohio / Sediment and Bioaccumulation Studies
- Howards Bay, Superior, Wisconsin / Sediment Remediation / Monitoring Program
- Upper St. Louis River / St Louis Bay / Howards Bay / Duluth, Minnesota and Superior, Wisconsin / Sediment Remediation / Monitoring Program

## Northeast / New England / Mid-Atlantic Region

ALS has provided sediment program analytical services for projects conducted in the Northeast / Mid-Atlantic Region. These services were conducted in accordance with national, regional and local regulatory and technical protocols, including: NJDEP Dredge Material Management Technical Manual, *Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters (1997)*; NJDEP Soil Remediation Residential Direct Contact Criteria (2008); NJDEP Ecological Screening Criteria Table (March 2009); PADEP Dredge Material Management Program; PADEP Land Recycling Program (Act 2); NYSDEC *Technical Guidance for Screening Contaminated Sediments (1998, 1999)*; and related requirements. These projects have included analyses of sediment, tissue and water samples for a variety of trace metals, organics, and conventional chemical constituents. Highlights of ALS regional sediment program experience includes:

- Lower Passaic River, New Jersey Study Area / Remedial Investigation / Sediment, Water Quality, and Bioaccumulation Studies
- Gowanus Canal, New York / Remedial Investigation / Sediment Monitoring Program
- Lake Onondaga, New York / Remedial Investigation / Site Restoration / Sediment Studies
- Hudson River, New York / Site Remediation / Site Restoration / Sediment Studies
- Salem, New Jersey / Bioaccumulation Study for Lagoon Closure and Wetland Remediation
- Bayonne, New Jersey / Dredge Material Management / Underwater Cable Installation
- New Jersey / Dredge Material Management for Shoreline Stabilization
- Delaware River, Philadelphia, Pennsylvania / Container Ship Terminal / Maintenance Dredging / Sediment and Water Quality Studies
- U.S. Marine Corps Naval Support Facility (NSF) Indian Head, Maryland / Sediment and Bioaccumulation Studies
- Sprague Mill (Versailles Mill) Landfills and Ponds, Sprague Mill, Connecticut / Sediment and Bioaccumulation Studies
- Camp Peary Environmental Site 49F to Waller Mill Park and Reservoir, Norfolk, Virginia / Sediment and Bioaccumulation Studies
- Chesapeake Bay, Maryland / NOAA BioEffects and Status and Trends Studies
- Delaware River, New Jersey / Pennsylvania / NOAA BioEffects and Status and Trends Studies
- Delaware River Bay, Delaware / NOAA BioEffects and Status and Trends Studies

### **South / Southeast / Gulf of Mexico Region**

ALS has provided sediment program analytical services for projects conducted in the South / Southeast / Gulf of Mexico Region. These services were conducted in accordance with national, regional and local regulatory and technical protocols issued by: USACE, USEPA, USFWS, State Departments of Environmental Quality, State Departments of Wildlife and Fisheries, and Regional Port and Harbor Authorities. These projects have included analyses of sediment, tissue and water samples for a variety of trace metals, organics, and conventional chemical constituents. Highlights of ALS regional sediment program experience includes:

- Oil Spill Response / Remedial Investigation / Bioaccumulation Studies / Gulf of Mexico, Texas, Louisiana, Mississippi, Alabama, Florida, Port Arthur Texas / Remedial Investigation Program / Sediment Studies
- San Jacinto River Waste Pits and Houston Ship Channel, Texas / Waste Site Remediation / Sediment, Water Quality, and Bioaccumulation Studies
- Jacksonville, Florida Harbor / Maintenance Dredging / Sediment and Water Quality Studies
- Jacksonville Harbor, Florida / Ocean Dredged Material Disposal Site (ODMDS) / Sediment Bioaccumulation Studies
- San Juan, Puerto Rico / Harbor Maintenance Dredging / Sediment Bioaccumulation Studies
- LCP Chemical Site, Brunswick, Georgia / Sediment Investigation/Remediation / Sediment and Water Quality Studies

- Port Everglades, Florida / Harbor and Channel Deepening / Sediment Bioaccumulation Studies
- Pinellas County, Florida / Inlet Channel Maintenance Dredging / Beach Nourishment / Sediment and Water Quality Studies
- Naval Base Kings Bay, Georgia / Maintenance Dredging / Sediment and Water Quality Studies
- Okatibbee Lake, Collinsville, MS / Maintenance Dredging / Sediment and Water Quality Studies
- Mobile, Alabama; Gulfport Harbor, Mississippi; Pensacola, Florida / Ocean Dredged Material Disposal Sites / Sediment, Water Quality, and Bioaccumulation Studies
- Military Ocean Terminal Sunny Point (MOTSU), Southport, North Carolina / Maintenance Dredging / Sediment and Water Quality Studies
- Manatee Port and Harbor, Florida / Entrance Channel, Port, Harbor Deepening / Sediment Bioaccumulation Studies
- U.S. Navy Military Fuel Piers, Jacksonville, Florida / Maintenance Dredging / Sediment and Water Quality Studies
- Charleston, South Carolina / Harbor and Channel Maintenance Dredging / Sediment, Water Quality and Bioaccumulation Studies
- Canaveral Harbor, Trident Access Channel, Trident Turning Basin, Port Canaveral, Florida / Sediment Bioaccumulation Studies
- Miami, Florida / Harbor Expansion/ Maintenance Dredging / Sediment, Bioaccumulation Studies
- Blount Island Marine Terminal, Jacksonville Florida / Maintenance Dredging and Channel Deepening / Sediment and Water Quality Studies
- Atlantic Intracoastal Waterway (AIWW) / Channel Deepening and Maintenance Dredging / Sediment and Water Quality Studies
- Cape Fear, Wilmington, North Carolina / Sediment and Water Quality Studies
- Charleston, South Carolina Harbor / NOAA BioEffects and Status and Trends Studies
- Winyah Bay, South Carolina / NOAA BioEffects and Status and Trends Studies
- Galveston Bay, Texas / NOAA BioEffects and Status and Trends Studies
- Biscayne Bay, Texas / NOAA BioEffects and Status and Trends Studies
- Sabine Lake, Texas / NOAA BioEffects and Status and Trends Studies
- St. Lucie Bay, Florida / NOAA BioEffects and Status and Trends Studies

## XII. Quick Method Reference Guide (Commonly Required for Marine Projects)

### Physical/Geotechnical:

- Atterburg Limits (Liquid Limit / Plastic Limit) ASTM D4318
- Particle Size (Sieve only or Sieve /Hydrometer) ASTM D-422
- Particle Size (Sieve/Pipette) Puget Sound Estuary Protocol (PSEP)
- Specific Gravity ASTM D-854
- Solids, Total (TS) by ASTM D-2216, EPA 160.3M, or PSEP
- Solids, Total Volatile (TVS) by EPA 160.4M or PSEP

### Conventionals:

- Ammonia by EPA 350.1 M/Plumb
- Carbon, Total Organic (TOC) by 9060M, ASTM D-4129-82 M, Lloyd Kahn, Plumb, or PSEP
- Carbon, Organic Matter by Walkley Black
- Carbon, Speciated by Gustafsson, et al. or Laboratory SOP
- Sulfides, Total by EPA9030M/SM4500 or PSEP
- Sulfides, Acid Volatile (AVS) by EPA draft Method 1991
- Sulfides, Acid Volatile/Simultaneously Extracted Metals (AVS/SEM) by EPA draft Method 1991

### Metals and Speciated Metals:

- Arsenic Speciation by LC/ICP/MS or EPA 1632 for As(III), As(V), MMAs, DMAs
- Butyltins by Krone, et al. (GC/FPD)
- Hexavalent Chromium by EPA 7196, EPA 7199, EPA 218.6, or EPA 218.6-Low Level
- Mercury (CVAAS) by EPA 7470/7471
- Mercury Low Level (P&T/CVAFS) by EPA 1631
- Mercury, Methyl by EPA 1630 or 1630M
- Metals (ICP/OES) by EPA 6010 or 200.7
- Metals (ICP/MS) by EPA 6020, 200.8, or 1638
- Metals, Optional Sample Preparations (Combined w/Variou Instrumental)
  - EPA 1640 On-line Chelation
  - EPA 1640 Reductive Precipitation
  - EPA 3052M Total Dissolution
  - Laboratory SOP - Total Dissolution w/Chemical Separation
  - Laboratory SOP - Total Dissolution w/Fusion
  - Laboratory SOP - Dry Ash/Acid Soluble
- Metals, Total Dissolution by EPA 3052M or Laboratory SOP w/EPA 6020, 200.8, or 1638
- Metals, Various by EPA 7000 (GFAAS or FAAS)
- Selenium (GHAAS) by EPA 7741
- Selenium Speciation by LC/ICP/MS for Se(IV), Se(VI)

### Organics:

- Dioxins/Furans by HRGC/HRMS by EPA 8290 or 1613
- Endocrine Disrupting Compounds (EDCs) by LC/MS/MS
- Explosive Residue by EPA 8330 (8330B for Incremental Sampling)
- Explosive Residue–Low Level by LC/MS/M
- Lipids by EPA SW846/Gravimetric
- Lipids by Bligh–Dyer
- Nonylphenols (includes BPA) by ASTM D7065–06M
- PCB congeners (WHO / NOAA / NIST / USACE) by EPA 8082–LL
- PCB congeners (209/WHO/NOAA/NIST/USACE) by EPA 1668
- Perchlorate (IC) by EPA 314.0
- Perchlorate (LC/MS) by EPA 6850
- Perfluorinated Compounds (PFOA, PFOS, etc.) by LC/MS/MS
- Pharmaceutical Personal Care Products (PPCPs) by LC/MS/MS
- Phenols Low Level by EPA 8720/SIM or 8270/LVI
- Phthalates Low Level by EPA 8270/SIM or 8270/LVI
- Polyaromatic Hydrocarbons (PAHs) standard list by EPA 8270/SIM or 8270/LVI
- Polyaromatic Hydrocarbons (PAHs) Alkylated Homologs by EPA 8270/SIM
- Polybrominated Diphenyl Ethers (PBDE) and Polybrominated Biphenyls (PBB) by EPA 8270/SIM
- Polychlorinated Biphenyls (PCBs) Aroclors Low Level by EPA 8082–LL
- Organochlorine Pesticides Low Level by EPA 8081–LL
- Organochlorine Pesticides Low Level by EPA 1699M (GC/MS/MS)
- Organophosphorus Pesticides Low Level by EPA 8270/GC/MS/MS
- Semi-Volatile Organic Compounds (SVOC) by EPA 8270 or 8270–SIM

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