Investigation-Derived Waste Management

1. Purpose

This standard operating procedure describes the activities and responsibilities of the United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific with regard to management of investigation-derived waste (IDW). The purpose of this procedure is to provide guidance for the minimization, handling, labeling, temporary storage, inventory, classification, and disposal of IDW generated under the ER Program. This procedure will also apply to personal protective equipment (PPE), sampling equipment, decontamination fluids, non-IDW trash, non-indigenous IDW, and hazardous waste generated during implementation of removal or remedial actions. The information presented will be used to prepare and implement work plans (WPs) for IDW-related field activities. The results from implementation of WPs will then be used to develop and implement final IDW disposal plans.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

This procedure focuses on the requirements for minimizing, segregating, handling, labeling, storing, and inventorying IDW in the field. Certain drum inventory requirements related to the screening, sampling, classification, and disposal of IDW are also noted in this procedure.

3. Definitions

3.1 IDW

IDW consists of all materials generated during site investigations that might be contaminated with chemicals of concern. IDW might consist of many types of potentially contaminated materials, including but not limited to, PPE, disposable sampling and decontamination equipment, investigation-derived soil, sludge, and sediment, well development and purge water, and decontamination fluids.

3.2 PPE

PPE, as defined in this procedure, refers to all disposable materials used to protect personnel from contact with potentially contaminated site media, such as inner and outer gloves, Tyvek suits and overboots, and disposable respirator cartridges. Non-consumable items, such as steel-toe boots, respirators, and hard hats are not included in this procedure.

3.3 Disposable Sampling Equipment

Disposable sampling equipment consists of all single-use equipment that might have come in contact with potentially contaminated site media, including sample bailers, Draeger air monitoring tubes, used soil sampling trowels and spatulas, plastic drop cloths, plastic bags and bucket liners, and sample containers from field analytical test kits.

3.4 Investigation-Derived Soil, Sludge, and Sediment

Investigation-derived soil consists of all potentially contaminated soil that is disturbed as part of site investigation activities. The most commonly encountered form of IDW soil is drill cuttings brought to the ground surface by drilling. Other forms of disturbed soil, including trenching spoils and excess soil remaining from surface sampling, should not be stored as IDW. Excavated soil should be returned to its source if site conditions permit.

Investigation-derived sludge consists of all potentially contaminated sludge materials generated or disturbed during site investigation activities. Generated sludge might consist of drilling mud used or created during intrusive activities. Other sludge might include solvents or petroleum-based materials encountered at the bottom of storage tanks and grease traps.

Investigation-derived sediment consists of all potentially contaminated sediments that are generated or disturbed during site investigation activities. Generated sediments might include solids that settle out of suspension from well development, purge, or decontamination water (see Definitions 3.5 and 3.6) while stored in 55-gallon drums or during sample filtration. Disturbed sediments might also consist of catch basin sediments or excess sediment from surface water activities.

3.5 Well Development and Purge Water

Development water consists of groundwater withdrawn from newly installed monitoring wells in preparation for well purging or pump testing. Monitoring well development methods are discussed in Procedure I-C-2, *Monitoring Well Development*.

Purge water consists of groundwater that is removed from monitoring wells immediately prior to sampling. Well purging methods are discussed in Procedure I-C-3, *Monitoring Well Sampling*. Groundwater derived during aquifer testing shall be addressed on a site-specific basis. Procedures for handling groundwater generated during aquifer testing shall be included in the WP or equivalent document for the CTO.

3.6 Decontamination Fluids

Decontamination fluids consist of all fluids used in decontamination procedures conducted during site investigation activities. These fluids consist of wash water, rinse water, and solvents used for the decontamination of non-consumable PPE, sampling equipment, and drilling equipment. Decontamination procedures are discussed in Procedure I-F, *Equipment Decontamination*.

3.7 Non-IDW Trash

Non-IDW trash is all waste materials, such as waste paper, drink containers, food, and packaging, generated in the support zone that have not come in contact with potentially contaminated site media.

3.8 NON-INDIGENOUS IDW

Non-indigenous IDW consists of all waste materials from offsite sources that are generated in the transition or contamination reduction zones and have not come in contact with potentially contaminated site media. Non-indigenous IDW includes materials, such as PPE from "clean" field activities (e.g., field blank generation, water sampling events) and refuse from monitoring well installation (e.g., unused sections of well casing, used bentonite buckets, sand bags, and cement bags).

Non-indigenous waste does not include material/waste that is abandoned at the ER site (including the IDW waste storage area) by other parties not associated with the ER work. Disposal of abandoned material/waste in the vicinity of IDW is the responsibility of the property owner (e.g., Navy Region Hawaii) or party responsible for abandoning the material/waste. The ER contractor shall notify the Contracting Officer's Representative (COR) of the situation as soon as possible so that recovery actions can be coordinated by the Government.

3.9 RESOURCE CONSERVATION AND RECOVERY ACT (RCRA) HAZARDOUS WASTE

Under the RCRA, a solid waste that is not excluded from regulation is defined as hazardous if it:

- Is "listed" as a hazardous waste in Chapter 40, Code of Federal Regulations (CFR), Parts 261.31 through 261.33
- Exhibits any of four hazardous "characteristics"—ignitability, corrosivity, reactivity, or toxicity (as determined using the Toxicity Characteristic Leachate Procedure [TCLP]) (40 CFR 261.20-24)
- Is subject to certain "mixture" or "derived-from" rules (40 CFR 261.3).

Under certain circumstances, petroleum- or polychlorinated biphenyl (PCB)-contaminated wastes are not considered RCRA hazardous when they only exhibit toxicity characteristic (40 CFR 261.4(b)(10) and 261.8). If IDW is determined to be RCRA hazardous waste, then RCRA storage, transport, and disposal requirements shall apply unless exempt.

3.10 RCRA LAND DISPOSAL RESTRICTIONS (LDR)

Land disposal, as defined in RCRA, is any placement of RCRA hazardous waste on the land in a waste pile, landfill, impoundment, well, land treatment area, etc. LDRs are regulatory restrictions placed on land disposal, including pre-treatment standards, engineered containment, capacity constraints, and reporting and permitting requirements.

3.11 AREA OF CONTAMINATION (AOC)

The U.S. Environmental Protection Agency (EPA) considers the RCRA AOC to be a single land-based disposal unit, usually a "landfill," and includes non-discrete land areas in which there is generally dispersed contamination. Storing IDW in a container (i.e., portable storage devices, such as drums and tanks) within the AOC and returning it to its source, whether RCRA hazardous or not, does not trigger RCRA LDRs. In addition, sampling and direct replacement of wastes within an AOC do *not* constitute land disposal.

3.12 CERCLA HAZARDOUS SUBSTANCES

The Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) hazardous substances are listed in 40 CFR Table 302.4 and include substances regulated by the RCRA Subtitle C, Clean Water Act (CWA), Clean Air Act (CAA), and Toxic Substances Control Act (TSCA). The CFR is updated annually; therefore, the most recent CFR should be referenced for the CERCLA hazardous waste list.

CERCLA hazardous substances are defined independent of their concentration level (i.e., any detection of a listed CERCLA constituent is considered a "CERCLA hazardous substance"). "Reportable quantities" identified for chemicals in 40 CFR Table 302.4 concern only CERCLA and RCRA requirements for notification to EPA when a release has occurred; they do not dictate whether a chemical is a hazardous substance.

The definition of CERCLA hazardous substances excludes "petroleum, including crude oil or any fraction thereof;" natural gas; natural gas liquids; liquefied natural gas; and synthetic gas usable for fuel, unless specifically listed or designated under the act. Excluded fractions of crude oil contain hazardous substances, such as benzene, that are indigenous in those petroleum substances or that are normally mixed with or added to petroleum during the refining process. However, hazardous substances that are (1) added to petroleum after the refining process, (2) increase in concentration as a result of contamination of the petroleum during use, or (3) commingled with petroleum after a release to the environment, are not considered part of the petroleum exclusion provision, and therefore, are regulated under CERCLA. In addition, some waste oils are regulated under CERCLA because they are specifically listed.

The scope of CERCLA hazardous substances includes the smaller subsets of RCRA hazardous wastes, PCB Aroclors, and other constituents. Therefore, a RCRA hazardous waste is always considered a CERCLA hazardous substance for a CERCLA-driven response action; however, a CERCLA hazardous substance is not always a RCRA hazardous waste.

CERCLA only regulates releases or threats of releases of hazardous substances into the environment. If there is no evidence that (1) a release has occurred (based on site history, visual observations, background metals evaluation), (2) there is a threat of release (as from abandoned, discarded, or non-maintained chemical receptacles), or (3) the release has entered the environment (as defined below), then CERCLA does not regulate the constituent even though it is identified on the CERCLA hazardous substance list.

3.12.1 CERCLA Hazardous Substances: TSCA/PCBs

PCBs are a CERCLA hazardous substance. PCBs belong to a broad family of man-made organic chemicals known as chlorinated hydrocarbons. PCBs were domestically manufactured from 1929 until their manufacture was banned in 1979. They have a range of toxicity and vary in consistency from thin, light-colored liquids to yellow or black waxy solids. Due to their non-flammability, chemical stability, high boiling point, and electrical insulating properties, PCBs were used in hundreds of industrial and commercial applications including electrical, heat transfer, and hydraulic equipment; as plasticizers in paints, plastics, and rubber products; in pigments, dyes, and carbonless copy paper; and many other industrial applications. Although no longer commercially produced in the United States, PCBs may be present in products and materials produced before the 1979 PCB ban.

If PCBs are detected at concentrations equal to or greater than 50 parts per million (ppm), the sample is considered TSCA-regulated. Current PCB regulations can be found in the CFR at 40 761. The EPA Q and A Manual (EPA 2009), referring to CFR 761.61 explains PCB remediation waste must be managed and disposed of based on the concentration at which the PCBs are found. It is unacceptable to dilute the as-found concentration of the contaminated soil by mixing it with clean soil during excavation or other IDW management activities.

3.13 Environment

Environment means navigable waters, ocean waters, surface water, groundwater, drinking water supply, land surface or subsurface strata, and ambient air, within the U.S. or under federal jurisdiction (see Section 101(8) of CERCLA or 40 CFR 300.5 for complete definition).

3.14 Onsite Area

The CERCLA onsite area is defined in 40 CFR 300.400(e)(1) as an area that includes:

- AOC
- All suitable areas in very close proximity to the contamination that are necessary for the implementation of the response action

The delineation of the onsite area is further discussed in Volume 55 Federal Register (FR) Page 8688 and EPA guidance.

Neither CERCLA, the National Oil and Hazardous Substances Pollution Contingency Plan, nor RCRA define the terms "area of contamination" or "contamination." However, the area of contamination is interpreted as containing "varying types and concentrations of contaminants" (55 FR 8760) that may or may not pose a risk to human health or the environment.

The onsite area may also include several noncontiguous aerial extents of contaminations if they share a common nexus (55 FR 8690).

3.15 Offsite Area

The offsite area consists of all areas outside the onsite area.

3.16 CERCLA Offsite Rule

The CERCLA offsite rule (400 CFR 300.440) states that IDW containing CERCLA hazardous substances (at any concentration) must be stored, treated, or disposed of offsite only at facilities having current EPA approval to accept such CERCLA wastes. RCRA-permitted facilities (Subtitle C and D) must also have specific EPA approval to accept waste generated at a CERCLA site (even if the waste is RCRA hazardous).

With some restrictions, the offsite rule does not apply to the following:

- Wastes generated during non-CERCLA actions
- Treatability study samples

- Wastes generated during emergency response actions
- Laboratory samples

CERCLA allows IDW to be managed, stored, and disposed of onsite within or near the AOC without the need for EPA approval (i.e., CERCLA facility approval) or RCRA permits. If IDW is to be stored or disposed of on site, the onsite area (and the AOC) should be delineated on a figure in the project field book and revised, based on best professional judgment, as site data become available.

4. Responsibilities

The prime contractor CTO Manager is responsible for preparing WPs and IDW disposal plans and reports in compliance with this procedure, and is responsible for documenting instances of noncompliance. The CTO Manager is responsible for ensuring that all personnel involved in sampling and/or testing shall have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

The prime contractor QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

The Field Manager is responsible for implementing this IDW procedure and ensuring that all project field staff follow these procedures.

Field personnel are responsible for the implementation of this procedure.

5. IDW Management Procedures

The procedures for IDW management in the field are described below.

5.1 Planning for IDW Management

The project team should begin planning for IDW issues early in the site investigation planning stage. The proper management of IDW involves all of the following tasks:

- Obtain Navy approval for a designated IDW storage area prior to commencement of field work
  - Complete Navy form, including IDW Tracking Sheet and provide to remedial project manager (RPM) for processing
- Waste generation and minimization
- Chemical screening and characterization of the waste
- Waste handling, storage, and associated maintenance in compliance with all regulations (prepare an IDW drum inventory, ensure storage areas are compliant with type of waste [double containment, TSCA requirements, etc.] maintain condition of drum and labeling, maintain safety and assess controls, comply with permit requirements [for offsite storage])
- Waste transport and disposal within required holding times
- Waste tracking, documentation, record keeping, and reporting

As part of IDW planning, the CTO Manager should consult with the COR and environmental regulatory agencies to clearly identify the primary federal or state regulatory authority that is driving the site investigation. This authority may be CERCLA, RCRA (Subtitle C), RCRA (subtitle I), TSCA, CWA, or an equivalent state program. The primary investigation authority and regulations promulgated under this authority set forth requirements for IDW management. These requirements may differ under the various response authorities. For CERCLA-driven actions, IDW storage and disposal should comply with all applicable or relevant and appropriate requirements (ARARs) and to-be-considered (TBC) criteria to the extent practicable.

Lastly, the CTO Manager should consider the disposal criteria of the anticipated disposal facility when developing the sampling and analysis plan (SAP). Some offsite facilities do not accept waste that is characterized by association with samples collected from the investigation site or they may require analytical data for chemicals that are not of potential concern at the site. Facility disposal criteria may dictate laboratory reporting limits.

If unknown waste is observed onsite, notify the project RPM and COR for further instructions.

5.2 IDW MINIMIZATION

Field managers (FMs) and their designates shall minimize the generation of onsite IDW to reduce the need for special storage or disposal requirements that might result in substantial additional costs and provide little or no reduction in site risks (EPA 1992b). Reduce the volume of IDW by applying minimization practices throughout the course of site investigation activities. These minimization strategies include substitution of biodegradable raw materials; using low-volume IDW-generating drilling techniques; where possible, returning excess material to the source location; using disposable sampling equipment versus generating more decontamination fluids from reusable sampling equipment; using bucket and drum liners; and separating trash from IDW.

Material substitution consists of selecting materials that degrade readily or have reduced potential for chemical impacts to the site and the environment. An example of this practice is the use of biodegradable detergents (e.g., Alconox or non-phosphate detergents) for decontamination of non-consumable PPE and sampling equipment. In addition, field equipment decontamination can be conducted using isopropyl alcohol rather than hexane or other solvents (for most analytes of concern) to reduce the potential onsite chemical impacts of the decontamination solvent. Select decontamination solvents carefully so that the solvents, and their known decomposition products, are *not* potentially RCRA hazardous waste, unless absolutely necessary.

Give priority to drilling methods that minimize potential IDW generation. Select hollow-stem auger and air rotary methods, where feasible, over mud rotary methods. Mud rotary drilling produces waste drilling mud, while hollow stem and air rotary drilling methods produce relatively low volumes of soil waste. Use small-diameter borings and cores when soil is the only matrix to be sampled at the boring location; however, the installation of monitoring wells requires the use of larger-diameter borings.

If possible, return soil, sludge, or sediment removed from borings, containment areas, and shallow test trenches to the source immediately after sampling and/or geological logging of the soils (EPA 1991, 1992b). Immediate replacement of solid waste in the source location during investigation activities avoids RCRA LDRs, which permit movement of IDW within the same AOC without considering land disposal to have occurred, even if the IDW is later determined to contain RCRA

hazardous material (EPA 1991). Place soil IDW from borings and trenches on polyethylene sheeting (e.g., Visqueen) during excavation and segregate it by approximate depth and any apparent contamination (i.e., visible staining). Following excavation, replace the soil IDW from above the saturated layer into the boring or trench and compact it, if possible. Efforts should be made to return the waste to the approximate depth from which it was generated. Soil and sludge IDW generated at or below the saturated layer of a boring or trench should be placed in drums and not returned to the source area. Suspected contaminated soil and sludge IDW generated above the saturated layer of a boring or trench should not be returned below the saturated layer."

Often monitoring wells are constructed outside the area of concern for soil contamination to sample for potential groundwater contamination or collect characteristic background data. At these locations, soil cuttings generated from above the saturation zone may be immediately disposed of near the wellhead in a shallow pit covered with natural topsoil from the site, and compacted. Contain soil and sludge IDW generated at or below the saturated layer in drums.

Reduce the quantity of decontamination rinse water generated by using dedicated and disposable sampling equipment, such as plastic bailers, trowels, and drum thieves that do not require decontamination. In general, decontamination fluids, and well development and purge water should not be minimized because the integrity of the associated analytical data might be affected.

Minimize the storage of visibly soiled PPE and disposable sampling equipment IDW by implementing decontamination procedures. If, based upon the best professional judgment of the FM, the PPE and disposable sampling equipment can be rendered non-contaminated after decontamination, then double-bag the PPE and disposable sampling equipment and dispose of it off site at a (RCRA Subtitle D) municipal solid waste disposal facility at the end of each work day (EPA 1991, 1992b). Since the decontaminated waste does not contain CERCLA hazardous substances, it need not be disposed of at a CERCLA-approved disposal facility in accordance with the CERCLA offsite rule.

Bucket liners can be used in the decontamination program to reduce the volume of solid IDW generated, and reduce costs on larger projects. The plastic bucket liners can be crushed into a smaller volume than the buckets, and only a small number of plastic decontamination buckets are required for the entire project. The larger, heavy-duty, 55-gallon drum liners can be used for heavily contaminated IDW to provide secondary containment, and reduce the costs of disposal and drum recycling. Drum liners may extend the containment life of the drums in severe climates and will reduce the costs of cleaning out the drums prior to recycling.

All waste materials generated in the support zone are considered non-IDW trash. To minimize the total volume of IDW, separate all trash from IDW, seal it in garbage bags, and properly dispose of it off site as municipal waste at the end of each work day.

Keep excess cement, sand, and bentonite grout prepared for monitoring well construction to a minimum. FMs shall observe well construction to ensure that a sufficient, but not excessive, volume of grout is prepared. Some excess grout may be produced. Unused grout (that should not come in contact with potentially contaminated soil or groundwater) shall be considered non-hazardous trash, and the drilling subcontractor shall dispose of it off site. Surplus materials from monitoring well installation, such as scrap plastic sections, used bentonite buckets, and cement/sand bags that do not

come in contact with potentially contaminated soil, shall be considered non-IDW trash, the drilling subcontractor shall dispose of it off site.

Following proper segregation procedures, as discussed in the next section, can minimize the quantity of contaminated IDW generated.

5.3 SEGREGATION OF IDW BY MATRIX AND LOCATION

It is necessary to properly segregate IDW in order to:

- Avoid commingling contaminated waste with clean waste, thereby creating a larger volume of waste that must be treated as contaminated
- Facilitate the sampling, screening, classification, and disposal of waste that may require different management methods

Take efforts to segregate IDW even when these activities will increase storage container and storage space requirements. These efforts will drastically reduce the sampling and documentation required for characterizing the waste and their associated costs.

In general, segregate IDW by matrix and source location and depth at the time it is generated. IDW from only one matrix shall be stored in a single drum (e.g., soil, sediment, water or PPE shall *not* be mixed in one drum). Groundwater and decontamination water should not be commingled; however, development and purge water from the same well may be stored together.

In general, IDW from separate sources should not be combined in a single drum or stockpile. Take efforts to segregate waste by increments of depth below ground surface. Most importantly, segregate soil IDW generated at or from below the saturated zone from soil generated above this zone (soil below this zone might be impacted by contaminated groundwater, whereas soil above the zone may be "clean"). Similarly, segregate soil above and below an underground storage tank (UST). Label each drum of soil to indicate the approximate depth range from which it was generated; this task may require cuttings to be segregated on plastic sheeting as they are generated or drums to be filled during the trenching or boring operation if this can be done in a safe manner.

It is possible that monitoring well development and purge water will contain suspended solids, which will settle to the bottom of the storage drum as sediment. Include significant observations on the turbidity or sediment load of the development or purge water in the logbook see Procedure III-D, *Logbooks* and Section 5.5). To avoid mixed matrices in a single drum (i.e., sediment and water), it may be necessary to decant the liquids into a separate drum after the sediments have settled out. This segregation may be accomplished during subsequent IDW sampling activities or during consolidation in a holding tank prior to disposal.

Place potentially contaminated well construction materials in a separate drum. No soil, sediment, sludge, or liquid IDW shall be placed in drums with potentially contaminated waste well construction materials. In addition, potentially contaminated well construction materials from separate monitoring wells shall not be commingled.

Store potentially contaminated PPE and disposable sampling equipment in drums separate from other IDW. Segregate PPE from generally clean field activities, such as water sampling, from visibly

soiled PPE, double-bag it, and dispose of it off site as municipal waste. Disposable sampling equipment from activities, such as soil, sediment, and sludge sampling, includes plastic sheeting used as liner material in containment areas around drilling rigs and waste storage areas, disposable sampling equipment, and soiled decontamination equipment. If, according to the Field Manager's best professional judgment, the visibly soiled PPE can be decontaminated and rendered non-hazardous, then double-bag the decontaminated PPE and disposed of it off site as municipal waste (EPA 1991, 1992b). PPE and disposable sampling equipment generated on separate days in the field may be combined in a single drum, provided clean and visibly soiled IDW are segregated as discussed above.

IDW generated from the use of field analytical test kits consists of those parts of the kit that have come into contact with potentially contaminated site media, and used or excess extracting solvents and other reagents. Contain potentially contaminated solid test kit IDW in plastic bags and store it with contaminated PPE or disposable sampling equipment IDW from the same source area as soil material used for the analyses. Segregate the small volumes of waste solvents, reagents, and water samples used in field test kits, and dispose of it accordingly (based upon the characteristics of the solvents as described in this procedure). Most other test kit materials should be considered non-IDW trash, and be disposed of as municipal waste.

Store decontamination fluids in drums separate from groundwater and other IDW. If practical, decontamination fluids generated from different sources should not be stored in the same drum. If decontamination fluids generated over several days or from different sources are stored in a single drum, record information about the dates and IDW sources represented in the drum. Note this information in the field notebook, on the drum label (Section 5.4.3), and in the drum inventory (Section 5.5).

The FM and designated personnel should separate the liquid and sediment portions of the equipment decontamination fluid present in the containment unit used by the drilling or excavation field crew. The contents of this unit normally consist of turbid decontamination fluid above a layer of predominantly coarse-grained sediment. When the contents of the containment unit are to be removed for storage in IDW drums, the FM shall instruct the field crew to place as much of the liquid into drums as possible and transfer the remaining solids into separate drums. Note observations of the turbidity and sediment load of the liquid IDW in the field notebook, on the drum label (Section 5.4.3), and in attachments to the drum inventory (Section 5.5). It is likely that decontamination fluids will contain minor amounts of suspended solids that will settle out of suspension to become sediment at the bottom of IDW storage drums. As noted above, it may be necessary to segregate the drummed water from sediment during subsequent IDW sampling or disposal activities.

Documentation for waste storage containers should include IDW source and segregation information and be maintained as follows:

1. Field logbook should be updated, at least weekly, with all IDW drum additions – update storage area location map to include new drum position and drum number.

2. External drum log (hard copy and electronic copy) should be updated with each IDW drum addition (drum numbers, source, and generation date) and closure of drum (fill date).

5.4 DRUM FILLING, HANDLING, AND LABELING, AND INVENTORYING

Drum handling consists of those actions necessary to prepare an IDW drum for labeling. Drum labeling consists of those actions required to legibly and permanently identify the contents of an IDW drum.

5.4.1 Drum Filling

Each drum of solid IDW shall be completely filled, when possible. For liquid IDW, drums should be left with headspace of approximately 5 percent by volume to allow for expansion of the liquid and potential volatile contaminants.

5.4.2 Drum Handling

IDW shall be containerized using U.S. Department of Transportation-(DOT) approved drums. The drums shall be made of steel or plastic, have a 55-gallon capacity, be completely painted or opaque, and have removable lids (i.e., United Nations Code 1A2 or 1H2). Drums having removable lids with bung holes are preferred to facilitate verification of drum contents. Typically 55-gallon drums are used, however small drums may be used depending on the amount of waste generated. New steel drums are preferred over recycled drums. Recycled drums should not be used for hazardous waste, PCBs or other regulated shipments. For short-term storage of liquid IDW prior to discharge, double-walled bulk steel or plastic storage tanks may be used. For this scenario, consider the scheduling and cost-effectiveness of this type of bulk storage, treatment, and discharge system versus longer-term drum storage.

The Guam Environmental Protection Agency may require double-walled drums or other secondary containment for the storage of liquid IDW. For long-term IDW storage at other project locations, the DOT-approved drums with removable lids are recommended. Verify the integrity of the foam or rubber sealing ring located on the underside of some drum lids prior to sealing drums containing IDW liquids. If the ring is only partially attached to the drum lid, or if a portion of the ring is missing, select another drum lid with a sealing ring that is in sound condition.

To prepare IDW drums for labeling, wipe clean the outer wall surfaces and drum lids of all material that might prevent legible and permanent labeling. If potentially contaminated material adheres to the outer surface of a drum, wipe that material from the drum, and segregate the paper towel or rag used to remove the material with visibly soiled PPE and disposable sampling equipment. Label all IDW drums and place them on appropriate pallets prior to storage.

5.4.3 Drum Labeling

Proper labeling of IDW drums is essential to the success and cost-effectiveness of subsequent waste screening and disposal activities (see Attachment I-A-6-1 and Attachment I-A-6-2). Labels shall be permanent and descriptive to facilitate correlation of field analytical data with the contents of individual IDW drums. Label all IDW drums using the **three distinct labeling methods** described below to ensure durability of the information. These three methods are completing and affixing preprinted NAVFAC Pacific ER Program labels; marking information on drum surfaces with paint; and, affixing aluminum tags to the drum. **Use of the preprinted labels, painted labeling, and aluminum tags is *mandatory***. These methods are described below.

5.4.3.1 PREPRINTED LABELS

Complete **two** preprinted NAVFAC Pacific ER Program drum labels as described below and presented in Attachment I-A-6-1. Seal both labels in separate heavy-duty, clear plastic bags, or use permanent markers on weatherproof stickers, to prevent moisture damage.

1. Place one label on the outside of the drum with the label data facing outward. Affix the bag/sticker to the drum at the midpoint of the drum height using a sufficient quantity of adhesive tape (e.g., duct tape, packing/strapping tape) so the bag will remain on the drum as long as possible during storage.

2. Affix the second label (sealed as mentioned above) to the underside of the drum lid, sealing it inside the drum when the lid is replaced.

The use of two or more preprinted labels for outer IDW drum identification purposes should be considered as a short-term backup to the information on the aluminum tags discussed below.

Print the requested information legibly on the drum labels in black, indelible ink. Instructions for entering the required drum-specific information for each label field are presented below:

CTO: Enter the four-digit number of the CTO for the project during which the IDW was generated. Include any initial zeroes in the CTO number (e.g., CTO 0047).

Activity-Site: Enter the name of the Navy activity responsible for the project site (e.g., Naval Supply Center, Naval Facilities Engineering Command Hawaii) and the name of the site where the project is taking place (e.g., Orote, Landfill, Building [Bldg.] 18).

Drum#: Enter the drum identification number according to the convention described below.

(xxxx-AA-DMzzz);

Where:

xxxx	represents the four-digit CTO number
AA	represents the unique site identifier assigned by the CTO Manager for multiple site CTOs (e.g., for CTO 0047, OW denotes Old Westpac, OR denotes Orote)
DM	represents a *drum* identification number
zzz	the sequential drum number for the site, beginning with 001

Date Collected: Enter the date the IDW was generated and placed in the drum. If IDW was generated over a number of days, enter the start and end dates for the period.

Contents: Record the source identification number on the label. Enter a "√" in the box corresponding to the type of IDW placed in the drum. For "Soil" and "Water," use the line provided to record observations on the condition of the drum contents (e.g., diesel odor, high turbidity, specific liquid IDW type). Check "Solid Waste" for PPE and indicate that PPE is present in the drum. Check

"Other" for disposable sampling equipment and potentially contaminated monitoring well construction materials, and indicate the type of waste on the line provided.

Project Type: Enter a "√" in the box corresponding to the type of investigation. Choices are Remedial Investigation, RCRA Facility Inspection, UST, and Other. If "Other" is specified, indicate the type of project in the "Comments" area, as described below.

Comments: Enter any additional information regarding the drum contents that will assist individuals who will characterize and dispose of the contents of the drum. "Other" project types include Site Inspection, Feasibility Study, Removal/Remedial Action, and Emergency Response activity. In addition, use this space on the label to complete any descriptions that were too large to fit in preceding label fields, such as the turbidity of decontamination water or the site activities from which the PPE was generated.

For Information Contact: Enter the project COR activity / code, address, and phone number.

It is essential that all relevant information recorded on individual drum labels be repeated in the field notebook for later development of the drum inventory database (see Section 5.5 and Procedure III-D, *Logbooks*).

5.4.3.2 PAINTED LABELS

The second method for labeling drums is to paint label information directly on the outer surface of the drum. At a minimum, the information placed on the drum shall include the CTO number, the drum number (following the numbering convention given above), the source identification number and type, the generation date(s), and the telephone number provided at the bottom of the preprinted label appropriate for the project location. The drum surface shall be dry and free of material that could prevent legible labeling. Confine label information to the upper two-thirds of the total drum height. The top surface of the drum lid may be used as an additional labeling area, but this area should only be used *in addition* to the upper two-thirds of the sides of the drum. The printing on the drum shall be large enough to be easily legible. Yellow, white, black, or red paint markers (oil-based enamel paint) that are non-photodegradable are recommended to provide maximum durability and contrast with the drum surface.

5.4.3.3 ALUMINUM TAGS

The third method for labeling drums is to affix an aluminum tag to the drum with neatly printed information that shall consist of the **CTO number**, the **drum identification number**, the **type of contents**, the **generation date(s)**, the **source** identification number and type, and the **telephone number** provided at the bottom of the appropriate preprinted label. Attachment I-A-6-2 to this procedure presents an example of the aluminum tag, which shall measure approximately 1 inch by 3 inches, or larger. When a ballpoint pen is used to fill out the aluminum tag, the information is permanently recorded as indentations on the tag. A fine ballpoint pen shall be used, and block-printed lettering is required for legibility. Indentations on the tag shall be sufficiently deep to be legible after the label has been exposed to weathering for an extended period.

Complete aluminum tags after the drum has been sealed. Affix the tags to the drum using a wire, which passes through predrilled holes in the label and shall be wrapped around the bolt used to seal the drum lid. The wire is the most likely part of the aluminum tag to decay during exposure. Use of

plastic insulated, copper-core electrical wire of appropriate diameter is recommended if long-term exposure to severe weathering is anticipated.

5.4.3.4 WASTE LABELS

Standard green and white non-hazardous and/or other hazardous waste stickers may be used in conjunction with, but not in lieu of, the above labeling procedures.

5.5 DRUM INVENTORY

Accurate preparation of an IDW drum inventory is essential to all subsequent activities associated with IDW drum tracking and disposal. Prepare an inventory for each project in which IDW is generated, stored, and disposed of. This information provided in the inventory report constitutes the results of preparing and implementing an IDW sampling, screening, characterization, and disposal program for each site.

The drum inventory information shall include 10 elements that identify drum contents and indicate their outcome. These elements are discussed in Sections 5.5.1 through 5.5.10.

5.5.1 Navy Activity (Generator)/Site Name

Inventory data shall include the Navy activity and the site name where the IDW was generated (e.g., Fleet Industrial Supply Center Pearl/Red Hill, Naval Magazine Headquarters/USTs).

5.5.2 CTO Number

Inventory data shall include the four-digit CTO number associated with each drum (e.g., 0089) and contract number as necessary.

5.5.3 Drum Number

Include the drum number assigned to each drum in the inventory database. Drum numbers shall adhere to the numbering convention presented in Section 5.4.3.1 (e.g., 0091-LF-DM006).

5.5.4 Storage Location Prior to Disposal

Include the storage location of each drum prior to disposal in the inventory database (e.g., Bldg. 394 Battery Disassembly Area, or Adjacent to West end of Bldg. 54). As part of the weekly inventory, a site visit to the IDW storage location shall be performed to observe the condition of the drums and covers. Drums and covers are considered acceptable when the integrity of the drums and covers are structurally intact, drum identification is legible, and the location of the drum storage is secure. An unacceptable classification will require recommendations to remedy the unacceptable classification.

5.5.5 Origin of Contents

Specify the source identification of the contents of each IDW drum in the inventory database (e.g., soil boring number, monitoring well number, sediment sampling location, or the multiple sources for PPE- or rinse water-generating activities).

5.5.6 IDW Type

Inventory data shall include the type of IDW in each drum (e.g., soil, PPE, disposable sampling equipment, sludge, sediment, development water, steam cleaning water, decontamination rinse water).

5.5.7 Waste Volume

Specify the amount of waste in each drum in the inventory database as a percentage of the total drum volume or an estimated percentage-filled level (e.g., 95 percent maximum for liquid IDW).

5.5.8 Generation Date

Inventory data shall include the date IDW was placed in each drum. If a drum contains IDW generated over more than one day, the start date for the period shall be specified in dd-mmm-yy format. This date is *not* to be confused with a RCRA hazardous waste accumulation date (40 CFR 262).

5.5.9 Expected Disposal Date

Specify the date each drum is expected to be disposed of as part of the inventory in mmm-yy format. This date is for the Navy's information only and shall not be considered contractually binding.

5.5.10 Actual Disposal Date

The actual drum disposal date occurs at the time of onsite disposal, or acceptance by the offsite treatment or disposal facility. Enter this date in the drum inventory data base only when such a date is available in dd-mmm-yy format.

Information required to complete all 10 of the inventory elements for the monthly inventory report described above and summarized in Attachment I-A-6-3, will be located on the IDW labels or provided by the CTO Manager.

Actual disposition of the IDW drum contents will be provided to the Navy.

5.6 IDW CLASSIFICATION

In general, the CTO Manager should follow IDW classification guidance contained in the *Generic IDW Disposal Plans* for Hawaii and Guam (Ogden 1994, 1995) and EPA guidance (EPA 1991, 1992a). The IDW classification process consists of chemical screening and characterization of the waste.

Various federal and state laws and guidance contain requirements for IDW management (handling, storage, transport, disposal, and recordkeeping) based on the type(s) and concentrations of chemicals present in the waste. To ensure that IDW is managed in compliance with these requirements and to evaluate disposal options, the CTO Manager should

- Directly sample and analyze the IDW or associate it with historical data, observed site conditions, and/or samples collected on site at the source of the waste
- Screen the waste to identify the maximum concentrations of individual chemicals in, or associated with, the waste

- Screen waste constituents against chemical background data, if available
- Characterize the waste based on regulated groups of chemical constituents present in the waste
- Screen waste constituents against risk-based health criteria, ARARs, and TBC criteria for onsite disposal, or disposal facility criteria for offsite disposal

Each of the above steps is distinct and should be performed separately to avoid potential mistakes in the IDW classification process. The following subsections discuss these steps in greater detail.

5.6.1 IDW Sampling and Chemical Screening

IDW should be screened to identify chemicals present in the waste and their maximum concentrations. Screening may be facilitated by (1) directly sampling the waste, (2) associating the waste with analytical results from samples collected at the source of the IDW (e.g., a well boring), (3) visual observation of the waste, (4) historical activity data from the site, or (5) a combination of these methods (e.g., association with limited sampling). Composite sampling may be required if the unit volume of IDW is non-homogeneous. Data from samples collected directly from the IDW should take precedence over associated site sample data when making waste management decisions. Procedure I-D-1, *Drum Sampling* discusses methods for drum sampling.

Typically, IDW is screened for chemicals of potential concern at the site and against background data if available. If IDW is generated from outside the suspected AOC (e.g., soil cuttings from the installation of a background monitoring well), assume it is clean, and dispose of it accordingly.

The CTO Manager should consider the disposal criteria of any offsite disposal facility anticipated to be used when developing the SAP. Some offsite facilities do not accept waste that is characterized by association with samples collected from the investigation site or they may require analytical data for chemicals that are not of potential concern at the site. Direct sampling and analysis of the waste may be required for these other constituents. Some disposal facilities prefer to collect and analyze the samples themselves. In addition, disposal facility criteria may dictate laboratory reporting limits. When possible, the CTO Manager should coordinate sampling and data requirements with the disposal subcontractor and anticipated disposal facility. Such efforts may allow IDW sampling to be conducted while the field team is mobilized for the site investigation, rather than conducting a separate IDW sampling event later.

5.6.2 IDW Characterization

Various federal and state laws and guidance contain requirements for IDW management (handling, storage, transport, disposal, and recordkeeping) based on the particular constituent or *group(s) of chemical constituents* present in the waste. Therefore, to ensure that IDW is managed in compliance with these requirements, characterize IDW based on the chemical screening results to determine whether any of the following regulated constituents are present in the waste:

- Petroleum hydrocarbons (regulated by RCRA Subtitle I when released from a UST; see 40 CFR Part 280)
- Hazardous wastes (regulated by RCRA Subtitle C; see 40 CFR 261-299)
- Non-hazardous, solid wastes (regulated by RCRA Subtitle D; see 40 CFR 257-258)

- Hazardous substances and commingled petroleum (regulated by CERCLA; see 40 CFR 300.400 and 302.4)
- PCBs (regulated by TSCA; see 40 CFR 700)
- Asbestos (regulated by CAA for disposal; see 40 CFR 61, Subpart M)
- Radioactive wastes (regulated by the Nuclear Regulatory Commission; see 10 CFR [various parts], 40 CFR, Subchapter F, and other applicable laws)

EPA regulations and guidance do not require IDW to be tested to properly characterize it. Instead waste may be characterized based on historical site data, site observations, analytical data from the source of the IDW, and professional judgment (EPA 1991). Specifically, the EPA has indicated that IDW may be assumed not to be "listed" wastes under RCRA unless available information about the site suggests otherwise (53 FR 51444). Similarly, RCRA procedures for determining whether waste exhibits RCRA hazardous characteristics do not require testing if the decision can be made by "applying knowledge of the hazard characteristic in light of the materials or process used" (40 CFR 262.11(c); EPA 1991). If applicable, the disposal plans and reports should state, "there is no evidence based on site data and observations that the IDW contains listed RCRA wastes or exhibits RCRA characteristics."

For soil IDW, the potential for exhibiting toxicity may be determined by comparing constituent concentrations in the waste against screening values that are 20 times the TCLP criteria as specified in Section 1.2 of EPA Method Solid Waste-846 1311 *Toxicity Characteristic Leaching Procedure* (EPA 2007). Otherwise, samples associated with the soil can be tested using the TCLP.

5.7 IDW STORAGE

In general, the CTO Manager should follow IDW storage guidance contained in the *Generic IDW Disposal Plans* for Hawaii and Guam (Ogden 1994, 1995) and EPA guidance (EPA 1990, 1991, 1992a).

Always store IDW in a manner that is secure, protected from weather, and protective of human health and the environment. It is preferable to store IDW within the AOC(s) or on site; however, the Navy may assign a specific IDW storage area away from the project site.

If the IDW is determined to be RCRA hazardous, then RCRA storage, transport, and disposal requirements may apply, including a limited **90-day** storage permit exemption period prior to required disposal. If onsite disposal is an option, store RCRA waste within the AOC so that RCRA LDRs will not apply in the future. LDRs may be triggered if the waste is stored within the onsite area, but outside of the AOC or if the waste is removed from and later returned to the AOC for disposal. The AOC concept does not affect the approach for managing IDW that did not come from the AOC, such as PPE, decontamination equipment and fluids, and groundwater. If RCRA hazardous, these wastes must be managed under RCRA and drummed and disposed of off site (EPA 1991).

RCRA waste should not be stored within the AOC prior to disposal when professional judgment suggests the IDW might pose an immediate or permanent public endangerment (EPA 1991b).

Offsite storage of CERCLA waste must comply with the CERCLA offsite rule (40 CFR 300.440).

If the IDW is determined to be TSCA-regulated, then TSCA storage requirements as described in CFR 764.65, transport, and disposal requirements apply, including a limited **30-day** storage period prior to required disposal. Storage requirements are as follows:

1. Storage facilities must provide an adequate roof and walls to prevent rain water from reaching the stored PCBs.

2. Storage facilities must provide an adequate floor that has continuous curbing with a minimum 6-inch-high curb.

3. Storage facilities must contain no drain valves, floor drains, expansion joints, sewer lines, or other openings that would permit liquids to flow from the curbed area.

4. Storage facilities must provide floors and curbing constructed of continuous smooth and impervious materials to minimize penetration of PCBs.

5. Storage facilities must not be located at a site that is below the 100-year flood water elevation.

6. PCBs in concentrations of 50 ppm or greater must be disposed of within 1 year after being placed in storage.

PCB waste can also be stored in a RCRA-approved waste storage area for 30 days from date of generation.

NAVFAC Pacific requires that all CERCLA, RCRA, and other types of waste be removed from JBPHH areas within 90 days of its generation, particularly within the shipyard area, and 30 days of generation for TSCA waste. Efforts should also be made to dispose of IDW within the 30- and 90-day periods at other Navy installations, unless the IDW will be managed with remediation waste to be generated during a cleanup action in the near future. The Navy may approve extensions of the storage time limit for wastes that are non-hazardous on a project-specific basis.

5.7.1 Drum Storage

Implement drum storage procedures to minimize potential human contact with the stored IDW and prevent extreme weathering of the stored drums. Place all IDW drums upright on pallets before the drums are stored. RCRA storage requirements include the following: containers shall be in good condition and closed during storage; wastes shall be compatible with containers; storage areas shall have a containment system; and spills or leaks shall be removed as necessary.

Place all IDW drums generated during field activities at a single AOC or designated IDW storage area together in a secure, fenced onsite area to prevent access to the drums by unauthorized personnel. When a secure area is not available, place drums in an area of the site with the least volume of human traffic. At a minimum, place plastic sheeting (or individual drum covers) around the stored drums. Post signage at the IDW storage area stating that drums should not be removed from the area without first contacting the Navy COR.

Liquid IDW drums must be stored under secondary containment (either secondary containment pallets or handmade plastic sheeting/polyvinyl chloride frame containment) and all IDW drums (soil

and water) must utilize secondary containment when stored within 15 feet of a surface water body or storm drain inlet.

Drums from projects involving multiple AOCs shall remain at the respective source areas where the IDW was generated. IDW should not be transferred off site for storage elsewhere, except under rare circumstances, such as the lack of a secure onsite storage area.

Implement proper drum storage practices to minimize damage to the drums from weathering and possible human exposure to the environment. When possible, store drums in dry, shaded areas and cover them with impervious plastic sheeting or tarpaulin material. Make every effort to protect the preprinted drum labels from direct exposure to sunlight, which causes ink on the labels to fade. In addition, store drums in areas that are not prone to flooding. Secure the impervious drum covers appropriately to prevent dislodging by the wind. It may be possible to obtain impervious plastic covers designed to fit over individual drums; nonetheless, repeat the labeling information on the outside of these opaque covers.

Drums in storage shall be placed with sufficient space between rows of drum pallets and shall not be stacked, such that authorized personnel may access all drums for inspection. Proper placement will also render subsequent IDW screening, sampling, and disposal more efficient when individual drum removal is necessary. It is recommended that IDW drums be segregated in separate rows/areas by matrix (i.e., soil, liquid or PPE/other).

If repeated visits are made to the project site, inspect the IDW drums to clear encroaching vegetation, check the condition and integrity of each drum, secondary containment if applicable, check and replace aluminum tags as necessary, and replace or restore the tarpaulin covers.

5.7.2 IDW Stockpiles

Consider IDW stockpiling only when a very large quantity of IDW will be generated. Segregate stockpiled IDW, and inventory it by source location and depth to the extent practicable. Stockpiling and media mixing should not be used as methods to dilute chemical concentrations in the waste. Line stockpiles on the bottom, cover it with sturdy plastic, and locate it in areas where weather elements (e.g., wind, rainfall runoff) will not cause migration of the waste. Never dispose of liquid IDW on a stockpile; drum or store liquid waste in other appropriate containers. Follow applicable regulation and guidance when sampling stockpiled waste for characterization purposes.

5.8 IDW Disposal

Various methods and requirements for onsite and offsite disposal of IDW are discussed in the *Generic IDW Disposal Plans* for Hawaii and Guam (Ogden 1994, 1995) and EPA guidance (EPA 1990, 1991, 1992b). This section explains the disposal evaluation process and highlights some of the more important requirements for onsite and offsite IDW disposal options.

IDW sampling, characterization, and disposal analysis, particularly for onsite disposal, can be unexpectedly complex and require compliance with many different laws (that act as ARARs for IDW management and disposal). Before preparing the IDW disposal plan, compare estimated costs for onsite vs. offsite disposal. Offsite disposal may be more cost effective than devising and documenting the justification for onsite disposal when the quantity of IDW is small (less than 10 drums) and/or the waste fails the initial conservative screening against conservative risk-based

criteria. Also weigh cost savings against the policy preference of the EPA and State of Hawaii Department of Health to manage and dispose of IDW on site, when possible.

5.8.1 Onsite Disposal

In general, the EPA preference is to dispose of IDW on site when the disposal action:

- Does not pose an unacceptable long-term risk to human health and the environment
- Is in accordance with chemical-, location- and action-specific ARARs "to the extent practicable" (40 CFR 300.415(i); 55 FR 8756)
- Does not introduce contaminants into clean soil or other site media
- Does not mobilize or significantly increase concentrations of any hazardous constituents already present in the environment
- Is consistent with the final remedy planned for the site
- Takes into account any community concerns regarding waste storage and the disposal method

Base onsite disposal options on best professional judgment and available site-specific data. For some projects, it may be prudent to store the waste temporarily until additional site data become available (e.g., sample analytical data, preliminary risk-assessment results, AOC delineation, and establishment of background values). Factors to consider include, but are not limited to the following:

- The detected or suspected contaminants, their concentrations, and total volume of IDW
- Media potentially affected (e.g., groundwater drinking source)
- Background metals data for site media
- Site access, conditions, and potential receptors
- Current and future land use
- Public perceptions (especially if drum storage and/or disposal takes place in open view)
- Time limits for IDW storage
- Potential requirements to treat waste before disposing of it on site
- Lack of unpaved areas to disposed of waste on site
- Potential wind, erosion, runoff, or flood conditions that might cause offsite migration of disposed waste
- Proximity to the ocean, surface water, or environmentally sensitive habitats
- Natural attenuation processes
- Need for additional utility survey before excavating to backfill waste
- Need for land use controls required to limit exposure pathways (e.g., backfill waste, provide permanent security around site, replant site to prevent erosion)

Protection of human health can be evaluated by comparing chemical concentrations in the waste to the more conservative of EPA residential regional screening levels), environmental action levels, and chemical-specific ARARs and TBC criteria. Ecological receptors can be protected by screening the IDW against EPA ecological soil screening levels. Onsite disposal of surface and groundwater IDW can be evaluated by initially screening against EPA tap-water PRGs, State Safe Drinking Water Standards (maximum contaminant levels and non-zero maximum contaminant level goals), and/or State Surface Water Quality Standards. These criteria are not always ARARs for the disposal method or site conditions; however, they may be useful to affirmatively show that the disposal is protective. Alternatively, the IDW may be associated with human-health and eco-risk assessment results for the site if the onsite placement of IDW is consistent with exposure pathway assumptions made during the risk assessment (e.g., contaminated soil might not present an unacceptable health risk at depth, but could pose such a risk if disposed of at the ground surface).

In general, return IDW consisting of environmental media to or near its source, and return waste generated from depth to its original depth, if possible and approved by NAVFAC in advance. Bury all contaminated soil and water IDW to be disposed of on site below grade at a depth of at least 3 feet and cover it with clean soil to reduce the potential for future exposure to human and ecological receptors.

Dispose of non-indigenous IDW and contaminated decontamination fluids off site. The cleaning detergent Alconox, often used in the decontamination process, is itself non-hazardous and biodegradable. Small quantities of clean decontamination water containing Alconox may be disposed of to clean areas on site. If onsite disposal is appropriate for RCRA IDW, this waste should be disposed of within the AOC to avoid the need to comply with LDRs.

IDW from several non-contiguous onsite areas may be consolidated and disposed of at one of the areas, provided a nexus exists between the wastes generated and response projects (55 FR 8690-8691).

IDW may also be temporarily disposed of back to the AOC without detailed analysis or documentation if the waste will be addressed with other site contamination during a future response action and will not present a significant short-term threat to human health and the environment.

5.8.2 Offsite Disposal

If onsite disposal is not a viable option, dispose of the IDW at an appropriate offsite treatment and/or disposal facility. Offsite transport and disposal of IDW must comply with all applicable laws and criteria specific to the chosen disposal facility. These requirements may include, but are not limited to the following:

- RCRA LDRs
- RCRA waste storage permits and time limits
- National Pollutant Discharge Elimination System and sewer disposal criteria
- CERCLA offsite rule
- TSCA treatment requirements
- DOT hazardous material transport packaging, manifesting, and security provisions

- International Maritime Organization ocean transport rules
- Certifications and training for waste transport contractors
- State notification requirements when importing certain types of waste

The CERCLA offsite rule (40 CFR 300.440) requires that CERCLA waste be disposed of only at facilities specifically approved by the EPA to receive such waste for treatment, storage, or disposal. The acceptability status of a disposal facility can change quickly (e.g., if there is a release at the facility); therefore, the CTO Manager should contact the EPA Region 9 CERCLA Offsite Rule Coordinator no more than 60 days prior to disposal of the IDW to verify the facility's approval status. The offsite rule applies to any CERCLA-driven remedial or removal action involving the offsite transfer of waste containing hazardous substances regardless of the concentrations present.

RCRA hazardous waste manifests must always be signed by authorized Navy personnel. In some cases, the Navy may authorize contractors to sign non-hazardous manifests. Navy authorization to allow contractor signature of non-hazardous manifests shall be based upon a Navy review of the contractor's RCRA and DOT training records. In addition, the Navy shall always be allowed the opportunity to review/approve non-hazardous manifests and waste profiles prior to waste disposal efforts.

Disposal of liquid IDW into the Navy sanitary sewer shall occur only if first approved by the Navy. Requests for disposal to Navy facilities should be coordinated through the COR. Discharge to the public sewer system is discouraged and should occur only if approved by state and local government agencies.

5.9 RECORDS

The CTO Manager is responsible for completing and updating the site-specific IDW drum inventory spreadsheet and submitting it as needed, and reviewing the IDW disposal plan (IDW disposal paperwork).

FMs and designates are responsible for documenting all IDW-related field activities in the field notebook including most elements of the IDW drum inventory spreadsheet. The correct methods for developing and maintaining a field notebook are presented in Procedure III-D, *Logbooks*.

Guidance related to preparing an IDW disposal plan (if required) is presented in the *Generic IDW Disposal Plans* for Hawaii and Guam (Ogden 1994, 1995).

5.9.1 IDW Disposal Documentation

Upon receipt of analytical data from the investigation or from IDW-specific analytical data, the generator information request form will be completed and provided to the IDW subcontractor to begin IDW characterization. Completed IDW disposal paperwork received from the IDW subcontractor should be reviewed for accuracy prior to submitting for Navy review.

The CTO Manager is responsible for submitting backup documentation (actual site or drum sampling results) along with the IDW disposal paperwork to the Navy.

Navy-approved contractor personnel may sign non-hazardous waste IDW documentation. Hazardous waste IDW documentation must be signed by an authorized Navy Environmental Coordinator.

All manifests (non-hazardous and hazardous) must be tracked, and if completed manifests (signed by disposal facility) are not received within 30 days of initial transportation, then contractor must notify the RPM weekly of the shipping status (e-mail is acceptable). Hazardous waste must be disposed of within 45 days of initial transportation. If not, specific IDW transportation details must be supplied to the Navy in order to prepare and file an exception report.

TSCA-regulated waste must be physically destroyed and or buried within 1 year of generation (date placed in IDW drum). Disposal certificates should be provided by the waste facility to the IDW subcontractor and Navy contractor.

Following disposal of IDW, the CTO Manager should prepare a short IDW disposal report summarizing the disposal operation and appending any associated records (e.g., final drum log, waste profiles, transport manifests, bills of lading, disposal facility certifications). Minimal topics to include in the report:

- IDW inventory and storage
- IDW chemical screening and characterization
- IDW transport and disposal
- Manifests
- Drum storage photographs
- Site figure

6. Health and Safety

Field Personnel shall perform work in accordance with the current (or as contractually obligated) United States Army Corps of Engineers Safety and Health Requirements Manual EM-385-1-1 (USACE 2008) and site-specific health and safety plan.

7. References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual*. OPNAV Instruction 5090.1D. 10 January.

Environmental Protection Agency, United States (EPA). 1990. *Guidance on Remedial Actions for Superfund Sites with PCB Contamination*. EPA/540/G-90-007. OSWER 9355.4-01. Office of Solid Waste and Emergency Response. August.

———. 1991. *Management of Investigation-Derived Wastes During Site Inspections*. EPA-540-G-91-009. Office of Emergency and Remedial Response. May.

———. 1992a. *Guidance for Performing Site Inspections under CERCLA*. EPA/540/R-92/021. Office of Emergency and Remedial Response. September.

———. 1992b. *Guide to Management of Investigation-Derived Wastes*. Quick reference fact sheet. OSWER Dir. 9345.3-03FS. Office of Solid Waste and Emergency Response. January.

———. 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*. 3rd ed., Revision 6. Office of Solid Waste. November. On-line updates at: http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm.

———. 2009. *Revisions to the PCB Q and A Manual*. January.

Ogden Environmental and Energy Services Company, Inc. (Ogden). 1994. *Final Generic IDW Screening, Sampling, Analysis, and Disposal Plan for Various Guam Naval Installations*. Pearl Harbor, HI: Pacific Division, Naval Facilities Engineering Command. September.

———. 1995. Generic IDW Screening, Sampling, Analysis, and Disposal Plan for Various Hawaii Naval Installations. Pearl Harbor, HI: Pacific Division, Naval Facilities Engineering Command. April.

United States Army Corps of Engineers (USACE). 2008. *Consolidated Safety and Health Requirements Manual*. EM-385-1-1. Includes Changes 1–7. 13 July 2012.

Procedure I-C-2, *Monitoring Well Development*.

Procedure I-C-3, *Monitoring Well Sampling*.

Procedure I-D-1, *Drum Sampling*.

Procedure I-F, *Equipment Decontamination*.

Procedure III-D, *Logbooks*.

8. Attachments

Attachment I-A-6-1: IDW Drum Label

Attachment I-A-6-2: Drum Label – Aluminum Tag

Attachment I-A-6-3: Monthly IDW Drum Inventory Updates

Attachment I-A-6-1
IDW Drum Label

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IDW Drum Label

Contract #:

CTO #:

ACTIVITY SITE: _____

_____

DRUM # (_ _ _ _ - _ _ - D M _ _ _)

DATE COLLECTED

CONTENTS: (please ✓ and explain)

☐ Soil _____

☐ Water _____

☐ Solid Waste _____

☐ Other _____

PROJECT TYPE

☐ RI ☐ RFI ☐ UST ☐ Other

COMMENTS: _____

_____

_____

_____

_____

FOR INFORMATION CONTACT:

COR Activity/ Code:

Address:

Telephone:

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Attachment I-A-6-2
Drum Label - Aluminum Tag

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Drum Label - Aluminum Tag

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Attachment I-A-6-3
Monthly IDW Drum Inventory Updates

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Table I-A-6-1: Monthly IDW Drum Inventory Updates

Navy Activity / Site Name (Generator Site)	CTO Number (0bbb)	Drum Number (xxxx-AA-DMzzz)	Drum Storage Location	Origin of Contents (Source ID #)	IDW Type	Waste Volume (Fill level %)	Waste Generation Date (dd-Mon-yy)	Expected Disposal Date (Mon-yy)	Actual Disposal Date (dd-Mon-yy)
Inspector:									
Date of Inspection:									
NSC Pearl Harbor/ Landfill	0068	0068-LF-DM001	NSC, Bldg 7	SB-1	Soil Cuttings	100	16-Dec-92	Dec-93	N/A
		0068-LF-DM002	N/A	MW-1 MW-2 MW-3	Purge Water	75	20-Dec-92	Jul 93	26-Jul-93
		0068-LF-DM003	N/A	MW-1 MW-2 MW-3	Decon. Water	95	20-Dec-92	Jul-93	26-Jul-93
		0068-LF-DM004	NSC, Bldg.16	SB-1 SB-2 SB-3 SB-4 MW-1 MW-2 MW-3	PPE	50	16-Dec-92	Oct-93	N/A
NAVSTA Guam/ Drum Storage	0047	0047-DS-DM001	Hazmat Storage Area	SB-1 SB-2	Soil Cuttings	100	18-Feb-93	Sep-93	N/A

N/A Not Applicable

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Analytical Data Validation Planning and Coordination

1. Purpose

This standard operating procedure describes data validation planning and coordination for all United States Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific sampling projects involving data validation.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

3.1 Critical Samples

Critical samples are samples that are especially important for assessing exposure and/or risk at a particular site, or are key in identifying remedial options.

3.2 Data Quality Assessment Report

The data quality assessment report summarizes the QA/quality control (QC) evaluation of the data according to precision, accuracy, representativeness, completeness, and comparability relative to the Project Quality Objectives (PQOs). The report provides a quantitative and qualitative assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall usability.

3.3 Data Validation

Data validation is a process that determines the technical usability of analytical data by comparison with a set of performance criteria. The performance criteria are designed in a manner that will enable the data user to know if the set of data will meet the intended purpose.

3.4 Data Validation Strategy

The data validation strategy includes the percentage of data to be validated (e.g., 100 percent or a smaller percentage), all samples from an entire sample delivery group (SDG) versus selected samples from various SDGs, and whether samples for Level D validation will be identified in advance or only after critical or risk-driving results for the risk assessment have been identified.

3.5 DATA VALIDATION LEVELS

The level of data validation possible for a given set of samples is based on the level of data package provided by the laboratory. The three levels of data validation considered are Level B (requires a Level 2 data package), Level C (requires a Level 3 data package), and Level D (requires a Level 4 data package). These levels have been identified in previous standard operating procedures as Cursory (Level B), Standard (Level C), and Full (Level D). Description for the extent of each level of data validation is presented below and further in Procedure II-A, *Data Validation*.

3.6 RAW DATA

Raw data is information that has not been processed, formatted, or reduced for end use. Examples of raw data include gas chromatographs, instrument printouts, copies of log books, chemist worksheets, etc.

3.7 SAMPLE DELIVERY GROUP (SDG)

A SDG, or analytical batch, typically includes up to 20 field samples plus associated batch QC samples.

4. Responsibilities

The prime contractor CTO Manager shall ensure coordination between data validators and appropriate project personnel. The CTO Manager is responsible for critical sample selection. The project chemist, laboratory coordinator, or other designated person, shall coordinate with the data validation task leader.

The prime contractor QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

5. Procedures

An independent party who is not responsible for the generation of the data shall perform data validation. Section 5.1 discusses guidelines for selecting a data validation strategy, while Section 5.2 presents planning and coordination guidelines.

5.1 DATA VALIDATION STRATEGY SELECTION

Consult the Contracting Officer's Representative, any appropriate regulatory agencies, and any Federal Facilities Agreements when choosing a data validation strategy. Clearly define the proposed level of effort for data validation in the project work plan. Based on the data validation requirements identified in the project planning documents, the analytical data may undergo "Level B," "Level C," or "Level D" data validation or some combination of these validation levels.

Guidelines for the required level of effort for data validation is described below and further in Procedure II-A, *Data Validation*.

5.1.1 Amount of Raw Data Acquired

It is recommended to request and obtain from the laboratory all raw data generated for the project sample analyses. While not all of the raw data will likely be reviewed, it is more time-efficient and cost-effective to obtain the data at the time of analysis than to request the laboratory to provide them

at a later date. In addition, project chemists and risk assessors may use portions of the raw data to more fully evaluate analytical data. Attachment I-A-7-1 presents the laboratory analytical data reporting requirements that shall be followed for the NAVFAC Pacific Area of Responsibility.

For projects with quick turnaround time (TAT) requirements, one option is to receive results only for the quick TAT, while receiving the remaining data at the normal TAT. This will allow the laboratory more time to compile the entire data package. Consult project-specific PQOs to determine if this approach is feasible.

5.1.2 Level B Validation

Level B validation is the least intensive of the three levels of data validation and is appropriate for non-critical data. Level B validation consists of evaluating factors such as holding times, spike analyses, blank analyses, and field QC samples. Examples of analytical results evaluated under data review include data generated during compliance monitoring, field analytical testing, or investigation derived waste sampling.

5.1.3 Level C Validation

Level C validation is the intermediary of the three levels of data validation and is appropriate for critical samples used in decision making. Level C validation consists of evaluating factors such as holding times, instrument calibration, spike and blank analyses, and field QC samples. Level C validation may be performed on a percentage or all of the project data. The exact percentage of data to undergo Level C validation will depend on the project objectives. Examples of analytical results evaluated under Level C validation include data generated for risk assessments, removal action verification, remedial designs, etc.

5.1.4 Level D Validation

Level D validation is the most rigorous of the three levels of data validation and is appropriate for critical samples used in decision making. Level D validation consists of evaluating factors such as holding times, instrument calibration, spike and blank analyses, field QC samples, and raw data. Level D validation may be performed on a percentage or all of the project data. The exact percentage of data to undergo Level D validation will depend on the project objectives. Examples of analytical results evaluated under Level D validation include data generated for risk assessments, removal action verification, remedial designs, etc.

Depending on the objectives of the project, a representative portion of data shall be chosen for Level D validation by selecting random samples and analyses, or more practically, be selected by identifying certain representative SDGs. This may include selecting all samples and analyses from one of the first SDGs of field samples for Level D data validation, and also for SDGs with different matrices, subsequent phases of work/mobilizations, and for each laboratory if more than one is used.

Larger projects typically require lower frequencies of Level D validation than smaller projects. For example, a project with one SDG may require 100 percent Level D validation. For a CTO with five SDGs, the first SDG may require Level D validation with the remaining four SDGs validated at Level C.

If significant issues, as defined in the data validation procedures presented in Section II of this procedures manual, are noted during Level D validation, additional Level D validation above the

originally planned percentage may be warranted and should be proposed. Additionally, the first several SDGs validated should be evaluated and corrective actions taken immediately if issues are identified.

5.2 PLANNING AND COORDINATION

During the planning and cost estimating stage of a project, contact the data validation task leader. Discuss the level of quality control, data validation strategy, number of samples per method, number of SDGs, schedule, and due dates. Copy all planning documents to the data validation task leader when they are completed (draft and final).

Hardcopy data validation reports are typically required and electronic entry of data qualifiers and qualification codes may be required if an analytical database is used for data interpretation.

Continuing coordination is critical. Notify the data validation task leader of any changes to the sampling schedule, analytical plan, or number of samples. Inform the data validators as well as the laboratory of every change from the chain of custody/analytical request form in sample numbers and/or requested analyses. Communicate changes to analytical methods agreed upon with the laboratory to the data validation task leader.

A schedule, which is updated as needed, is necessary to track the status of data validation activities. The prime contractor QA Manager or Technical Director shall coordinate and set priorities between CTOs. Attachment I-A-7-2 is an example of a form that may be used by CTO personnel to track the data validation status of hardcopy data.

A cross-reference list of field QC samples associated with site samples is required to validate data. This list must be provided by field personnel or from the chain-of-custody logbook (Procedure III–E, *Record Keeping, Sample Labeling, and Chain-of-Custody*).

6. Records

Data validation reports generated by data validators shall include content discussed in Procedure II-A, *Data Validation* and be included as an appendix in the report and summarized in the report. Changes in the schedule, number of samples, or analytical plan shall be sent to the data validators verbally and in writing.

The data validation effort shall be summarized for inclusion as a section of the report. It may also be helpful to summarize the data validation results in the form of a data quality assessment report (DQAR). The DQAR should summarize the net results of data validation for each QC parameter evaluated. It is recommended that precision, accuracy, and percent completeness objectives also be presented in the report. This task could be conducted by the data validators, or by project staff more familiar with the PQOs. The content and format of the DQAR is discussed in Procedure II-S, *Data Quality Assessment Report*.

As part of the summary, the project personnel shall ensure that all data requested for analysis and validation were actually analyzed and validated. Identification of rejected data (and the reasons) may be the most critical results. Data that have been qualified from detections to nondetections, or data for which numerical values have changed significantly, are also important. The summary may focus

on the analytes and samples that are considered most critical for each project and include a summary of field QC results by field QC type.

7. Health and Safety

Not applicable.

8. References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Procedure II-A, *Data Validation*.

Procedure II-S, *Data Quality Assessment Report*.

Procedure III–E, *Record Keeping, Sample Labeling, and Chain-of-Custody*.

9. Attachments

Attachment I-A-7-1: DoD QSM Appendix DoD A Reporting Requirements

Attachment I-A-7-2: Example Hardcopy Data Validation Status Tracking Form

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Attachment I-A-7-1
DoD Quality Systems Manual Appendix DoD A Reporting Requirements

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APPENDIX DOD-A – REPORTING REQUIREMENTS

In the absence of client specified reporting criteria, the reporting requirements outlined below shall be used for hard-copy data reports or electronic versions of hard-copy data (such as pdf). They include mandatory requirements for all printed data reports, and requirements for data reports requiring third party data review or validation. Optional reporting requirements are those that may be required by a specific project, depending upon their needs. The following elements are required: cover sheet, table of contents, case narrative, analytical results, sample management records, and Quality Assessment/Quality Control (QA/QC) information. Information for third-party review may be required depending on project-specific requirements or the method being used.

1.0 Cover Sheet

The cover sheet shall specify the following information:

- Title of report (i.e., test report, test certificate);
- Name and location of laboratory (to include a point of contact, phone and facsimile numbers, and e-mail address);
- Name and location of any subcontractor laboratories, and appropriate test method performed (information can also be located in the case narrative as an alternative);
- Unique identification of the report (such as serial number);
- Client name and address;
- Project name and site location;
- Statement of data authenticity and official signature and title of person authorizing report release;
- Amendments to previously released reports that clearly identify the serial number for the previous report and state the reason(s) for reissuance of the report; and
- Total number of pages.

2.0 Table of Contents

Laboratory data packages shall be organized in a format that allows for easy identification and retrieval of information. An index or table of contents shall be included for this purpose.

3.0 Case Narrative

A case narrative shall be included in each report. The purpose of the case narrative is to:

- Describe any abnormalities and deviations that may affect the analytical results;
- Summarize any issues in the data package that need to be highlighted for the data user to help them assess the usability of the data; and
- Provide a summary of samples included in the report with the methods employed in order to assist the user in interpretation.

The case narrative shall provide (Information need not be repeated if noted elsewhere in the data package):

- A table(s) summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical, preparation, and clean-up methods were performed. If multiple laboratories performed analyses, the name and location of each laboratory **shall** be associated with each sample;
- A list of samples that were received but not analyzed;
- Date of samples received;
- Sample preservation or condition at receipt;
- A description of extractions or analyses that are performed out of holding times;
- A definition of all data qualifiers or flags used;
- Identification of deviations of any calibration standards or QC sample results from appropriate acceptance limits and a discussion of the associated corrective actions taken by the laboratory;
- Identification of multiple sample runs with reason(s) identified (e.g., dilutions or multiple cleanups);
- Identification of samples and analytes for which manual integration was necessary; and
- Appropriate notation of any other factors that could affect the sample results (e.g., air bubbles in volatile organic compounds (VOC) sample vials, excess headspace in soil VOC containers, the presence of multiple phases, sample temperature or pH excursions, and container type or volume).

4.0 Analytical Results

The results for each sample shall contain the following information at a minimum: (Information need not be repeated if noted elsewhere in the data package):

- Project name and site location;
- Field sample ID number as written on custody form;
- Laboratory sample ID number;
- Preparation batch number(s);
- Matrix (soil, water, oil, air, etc.);
- Date and time sample collected;
- Date and time sample prepared;
- Date and time sample analyzed;
- Method numbers for all preparation, cleanup, and analysis procedures employed;
- Analyte or parameter with the Chemical Abstracts Service (CAS) Registry Number if available;

- Sample aliquot analyzed;
- Final extract volume;
- Identification of analytes in which manual integration occurred, including the cause and justification;
- Analytical results with correct number of significant figures;
- Detection Limit, Limit of Detection, and Limit of Quantitation associated with sample results and adjusted for sample-specific factors (e.g., aliquot size, dilution/concentration factors, and moisture content);
- Any data qualifiers assigned;
- Concentration units;
- Dilution factors;
- All multiple sample run results shall be reported;
- Percent moisture or percent solids (all soils are to be reported on a dry weight basis); and
- Statements of the estimated uncertainty of test results (optional).

5.0 Sample Management Records

Sample Management records shall include the documentation accompanying the samples, such as:

- Chain-of-custody records;
- Shipping documents;
- Records generated by the laboratory which detail the condition of the samples upon receipt at the laboratory (e.g., sample cooler receipt forms, cooler temperature, and sample pH);
- Telephone conversation or e-mail records associated with actions taken or quality issues; and
- Records of sample compositing done by the laboratory.

6.0 QA/QC Information

The minimum laboratory internal QC data package shall include:

- Method blank results;
- Percent recoveries for Laboratory Control Sample (LCS), Laboratory Control Sample Duplicates (LCSD), Matrix spike (MS), and Matrix Spike Duplicates (MSD);
- MSD or matrix duplicate Relative percent differences (RPD);
- Surrogate percent recoveries;
- Tracer recoveries;
- Spike concentrations for LCS, MS, surrogates;
- QC acceptance criteria for LCS, MS, surrogates;
- Post-Digestion Spike (PDS) recoveries;

- In-house or project specified LCS control limits, as applicable;
- Serial dilutions (SD) percent difference; and
- Batch numbers (preparation, analysis, and cleanup).

7.0 Data Reports for Third Party Review or Validation

When third party review or data validation is to be performed, the extent (stage) of data validation that can be performed is dependent upon the type (level) of data report delivered by the laboratory. The data report level and data validation stage required to meet project data quality objectives should be specifically defined in the QAPP.

The minimum reporting requirements for each level of data report are outlined below.

- A cover sheet, table of contents, and case narrative including all of the information specified in the above sections are required for all levels of data reports.
- **Level 1**: Analytical results, Sample Management Records.
- **Level 2**: **Level 1** reporting requirements plus QA/QC Information, Instrument QA/QC Information, Instrument and Preparation logs.
- **Level 3**: **Level 2** reporting requirements plus Instrument Quantitation Reports.
- **Level 4**: **Level 3** reporting requirements plus Instrument Chromatograms and Spectra.
- In addition, Standards traceability should be included in Levels 3 and 4 if a legal chain of custody is required.

The data validation guidelines established in other Department of Defense guidance or project-specific guidelines may have distinct reporting formats. The appropriate QAPP should be consulted to determine what type of data package is required.

Attachment I-A-7-2
Example Data Validation Status Tracking Form

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Table I-A-7-2-1: CTO xxxx Data Validation Report Status Tracking Form

SDG	Due Date	VOCs Rec'd	PCBs Rec'd	TPH Rec'd	Metals Rec'd	Cr+6 Rec'd	Otin Rec'd	TOC Rec'd
DB360	7/30	7/21	8/21	8/21	8/7	X	8/23	5/25
DB383	7/30	7/21	8/21	8/21		X	8/23	5/25
DB401	6/15	6/9	6/9	6/9	6/9	X	7/7	6/9
DC160	8/15	7/21	8/21	8/21			X	8/7
DC180	8/15	7/21	8/21	7/23		7/21	8/23	8/21
CK0693	7/30	X	X	X	X	7/20	X	X
CK0694	7/30	X	X	X	X	7/20	X	X
CK0732	7/30	X	X	X	X	7/20	X	X
DC205	9/15		X			X	X	
DC209	9/15		X			X	X	
DB429	9/15		X			X	X	
DB439	9/15		X			X	X	X
DB458	9/15		X			X	X	X

PCB polychlorinated biphenyl
TOC total organic carbon
TPH total petroleum hydrocarbons
VOC volatile organic compound
7/21 date data validation report was received
X no analysis for that method for that SDG
blank data validation report not yet received

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Sample Naming

1. Purpose

This standard operating procedure describes the naming convention for samples collected and analyzed, and whose resulting data will be stored in the database for the United States Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific projects. Unique sample names are used to facilitate tracking by laboratory personnel and project personnel, and for purposes of storing, sorting, and querying data in the database.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

3.1 Chain of Custody Sample Number

The chain of custody (COC) sample number is a five-character identification number that is used by the laboratory and project personnel for tracking purposes. A unique COC sample number must be used for each sample collected from a particular location at a particular time. It is useful for the first two characters to be letters unique to a particular site or project, while the remaining three characters may be digits from 001 to 999 (e.g., AA001). The COC sample number is the only identifier that should be presented to the laboratory.

3.2 Sample Identification Number

The sample identification number is a unique multi-alpha, multi-numeric identifier that is used by the field team to associate sampling results to the particular sampling location, sample type, number of times the location has been sampled, and depth. To avoid potential bias in sample analysis, the sample identifier is not provided to the laboratory. The sample identification number shall be recorded in the field logbook concurrently with the COC sample number.

4. Responsibilities

The prime contractor CTO Manager shall ensure that a proper sample naming convention is identified in the field sampling plan. The Field Quality Control (QC) Supervisor or other field-sampling leader shall ensure that the sample naming convention is implemented. The laboratory coordinator, CTO Manager, and/or other designated personnel shall ensure on a daily basis that unique, appropriate COC sample numbers and sample identifiers have been assigned. The prime

contractor QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

The prime contractor Technical Director will designate one person in each office (e.g., the laboratory coordinator) to track site designations used in the COC sample number.

5. Procedures

A COC sample number and sample identifier shall be assigned as described below. It is critical that each sample name have a unique COC sample number and sample identifier; otherwise, data cannot be properly stored and tracked in the database.

5.1 COC Sample Number

Use the following format for the COC sample number:

abccc

Where:

a = A letter indicating the office managing the CTO

b = A letter indicating the project or site, for example

- A = first site
- B = second site
- C = third site, etc.

ccc = Chronological number, for example

- 001 = first sample from the site
- 002 = second sample from the site
- 105 = 105th sample from the site
- Field QC samples should be included in this chronological sequence

For example, the 23rd sample from the Carpentry Shop Dip Tank site (assigned project "A" for b above; the office will be assigned "D") being investigated would be referred to as "DA023." This might be a soil sample, water sample, trip blank, equipment blank, field duplicate, or other sample type. Using this COC sample number, the samples will be submitted to the laboratory "blind," that is, the laboratory should not know whether each sample received is a site or field QC sample.

If a sample is lost during shipping, the replacement sample must be assigned a new COC sample number. If different containers for the same sample are shipped on different days, a new COC sample number must be assigned.

When numbering reaches the letter Z, the 26th site, it may begin with a new first letter "a," which must be coordinated with the prime contractor QA Manager or Technical Director and Coordinator or designee to ensure that it has not been used by another CTO.

Alternatively, the "ab" designators can serve to identify a unique project field, such as "RH" for the Red Hill site.

5.2 SAMPLE IDENTIFICATION NUMBER

The following format is provided as a suggested guidance. Individual site objectives may necessitate variations to the suggested guidance. Coordinate with the prime contractor QA Manager or Technical Director when considering deviating from this guidance.

AA-bbcc-dee-Dff.f

Where:

AA = Designates the site identification

bb = Sample type and matrix (see Table I-A-8-1)

cc = Location number (e.g., 01, 02, 03)

d = Field QC sample type (see Table I-A-8-2)

ee = Chronological sample number from a particular sampling location (e.g., 01, 02, 03)

D = The letter "D" denoting depth

ff.f = Depth of sample in feet bgs (to the measured decimal place). For field blanks, trip blanks and equipment blanks, the depth field will contain the month and date of collection.

For example, the first subsurface soil sample collected from the Foundry Building (FB) borehole location four at a depth of 10 feet would be designated "FB-BS04-S01-D10.0." These characters will establish a unique sample identifier that can be used when evaluating data.

Table I-A-8-1 presents the character identifiers to be used in the sample and matrix portion of the sample identification number. In all cases, the second letter indicates the sample matrix. Note grab, composite, and undisturbed sample designations in the field logbook.

Table I-A-8-1: Sample Type and Matrix Identifiers

Identifier	Sample Type	Matrix
SS	Surface Soil	Soil
IS	Surface Soil (ISM)	Soil
IB	Subsurface Soil (ISM)	Soil
BS	Subsurface Soil	Soil
BG	Subsurface Soil (Geotechnical)	Soil
SD	Sediment	Sediment
GW	Groundwater	Water
SW	Surface Water	Water
FP	Free Product	Oil
WQ	Water Blanks	Water
SG	Soil Gas	Soil gas
CC	Concrete Chips	Concrete

Identifier	Sample Type	Matrix
WS	Waste (IDW)	Soil
WW	Waste (IDW)	Water

IDW investigation-derived waste
ISM incremental sampling methodology

Table I-A-8-2 describes the field QC designator types. These field QC designators clarify the type of sample collected.

Table I-A-8-2: Field QC Sample Type Identifiers

Identifier	QC Sample Type	Description
S	Normal (Primary) Sample	All non-field QC samples
D	Duplicate	Collocate (adjacent liners)
R	Triplicate	Replicate
E	Equipment Rinsate	Water
B	Field Blank	Water
T	Trip Blank	Analytical-laboratory-prepared sample -Water
M	Trip Blank	Analytical-laboratory-prepared sample – Methanol
L	Batch Test Sample	Batch Test Leaching Model Sample
P	Blind Spike	Performance testing sample

6. Records

Sample identifiers (and COC sample numbers, if appropriate) shall be identified in advance if the exact numbers of samples to be collected are known; these numbers may be listed on a spreadsheet along with requested analyses to be used as a reference by field sampling personnel.

The COC/analytical request form must be used to track all sample names. Copies of each COC form shall be sent daily to the CTO Laboratory Coordinator and with the samples to the analytical laboratory. An example of a COC form is included as Attachment III-E-2 of Procedure III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody*.

In the field, personnel shall record in the field logbook the COC sample number of each sample collected, as well as additional information, such as the sampling, date, time, and pertinent comments.

7. Health and Safety

Not applicable.

8. References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Procedure III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody*.

9. Attachments

None.

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Monitoring Well Sampling

1. Purpose

This standard operating procedure describes the monitoring well sampling procedures to be used by United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific personnel.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

None.

4. Responsibilities

The prime contractor CTO Manager is responsible for ensuring that these standard groundwater sampling activities are followed during projects conducted under the NAVFAC Pacific ER Program. The CTO Manager or designee shall review all groundwater sampling forms on a minimum monthly basis. The CTO Manager is responsible for ensuring that all personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

The prime contractor QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

The Field Manager is responsible for ensuring that all project field staff follow these procedures.

Field sampling personnel are responsible for the implementation of this procedure.

Minimum qualifications for sampling personnel require that one individual on the field team shall have a minimum of 1 year experience with sampling monitoring wells.

The field sampler and/or task manager is responsible for directly supervising the groundwater sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling. If deviations from the procedure are required because of

anomalous field conditions, they must first be approved by the QA Manager or Technical Director and then documented in the field logbook and associated report or equivalent document.

5. Procedures

5.1 PURPOSE

This procedure establishes the method for sampling groundwater monitoring wells for water-borne contaminants and general groundwater chemistry. The objective is to obtain groundwater samples of aquifer conditions with as little alteration of water chemistry as possible.

5.2 PREPARATION

5.2.1 Site Background Information

Establish a thorough understanding of the purposes of the sampling event prior to field activities. Conduct a review of all available data obtained from the site and pertinent to the water sampling. Review well history data including, but not limited to, well locations, sampling history, purging rates, turbidity problems, previously used purging methods, well installation methods, well completion records (including depth of screened interval), well development methods, previous analytical results, presence of an immiscible phase, historical water levels, and general hydrogeologic conditions.

Previous groundwater development and sampling logs give a good indication of well purging rates and the types of problems that might be encountered during sampling, such as excessive turbidity and low well yield. They may also indicate where dedicated pumps are placed in the water column. To help minimize the potential for cross-contamination, well purging and sampling, and water level measurement collection shall proceed from the least contaminated to the most contaminated as indicated in previous analytical results. This order may be changed in the field if conditions warrant it, particularly if dedicated sampling equipment is used. A review of prior sampling procedures and results may also identify which purging and sampling techniques are appropriate for the parameters to be tested under a given set of field conditions.

5.2.2 Groundwater Analysis Selection

Establish the requisite field and laboratory analyses prior to water sampling. Decide on the types and numbers of QA/quality control (QC) samples to be collected (Procedure III-B, *Field QC Samples [Water, Soil]*), as well as the type and volume of sample preservatives, the number of sample containers (e.g., coolers), and the quantity of ice or other chilling materials. The sampling personnel shall ensure that the appropriate number and size sample containers are brought to the site, including extras in case of breakage or unexpected field conditions. Document the analytical requirements for groundwater analysis in the project-specific work plan.

5.3 GROUNDWATER SAMPLING PROCEDURES

Groundwater sampling procedures at a site shall include: (1) measurement of well depth to groundwater; (2) assessment of the presence or absence of an immiscible phase; (3) assessment of purge parameter stabilization; (4) purging of static water within the well and well bore; and (5) obtaining a groundwater sample. Each step is discussed in sequence below. Depending upon specific field conditions, additional steps may be necessary. As a rule, at least 24 hours should separate well development and well sampling events.

5.3.1 Measurement of Static Water Level Elevation

Measure the depth to standing water and the total depth of the well to the nearest 0.01 foot to provide baseline hydrologic data, to calculate the volume of water in the well, and to provide information on the integrity of the well (e.g., identification of siltation problems). Mark each well with a permanent, easily identified reference point for water level measurements whose location and elevation have been surveyed.

Before purging the well, measure water levels in all of the wells within the zone of influence of the well being purged. Measure water levels twice in quick succession and record each measurement. This will provide a water level database that describes water levels across the site at one time (a synoptic sampling). Measure the water level in each well immediately prior to purging the well.

The device used to measure the water level surface and depth of the well shall be sufficiently sensitive and accurate in order to obtain a measurement to the nearest 0.01 foot reliably. An electronic water level meter will usually be appropriate for this measurement; however, when the groundwater within a particular well is highly contaminated, an inexpensive weighted tape measure can be used to determine well depth to prevent adsorption of contaminants onto the meter tape. The presence of light, non-aqueous phase liquids (LNAPLs) and/or dense, non-aqueous phase liquids (DNAPLs) in a well requires measurement of the elevation of the top and the bottom of the product, generally using an interface probe. Water levels in such wells must then be corrected for density effects to accurately determine the elevation of the water table.

5.3.2 Decontamination of Equipment

Establish a decontamination station before beginning sampling. The station shall consist of an area of at least 4 feet by 2 feet covered with plastic sheeting and be located upwind of the well being sampled and far enough from potential contaminant sources to avoid contamination of clean equipment. The station shall be large enough to fit the appropriate number of wash and rinse buckets, and have sufficient room to place equipment after decontamination. One central cleaning area may be used throughout the entire sampling event. The area around the well being sampled shall also be covered with plastic sheeting to prevent spillage. Further details are presented in Procedure I-F, *Equipment Decontamination*.

Decontaminate each piece of equipment prior to entering the well. Also conduct decontamination prior to sampling at a site, even if the equipment has been decontaminated subsequent to its last usage. This precaution is taken to minimize the potential for cross-contamination. Additionally, decontaminate each piece of equipment used at the site prior to leaving the site. It is only necessary to decontaminate dedicated sampling equipment prior to installation within the well. Do not place clean sampling equipment directly on the ground or other contaminated surfaces prior to insertion into the well. Dedicated sampling equipment that has been certified by the manufacturer as being decontaminated can be placed in the well without onsite decontamination.

5.3.3 Detection of Immiscible Phase Layers

Complete the following steps for detecting the presence of LNAPL and DNAPL, as necessary, before the well is evacuated for conventional sampling:

1. Sample the headspace in the wellhead immediately after the well is opened for organic vapors using either a photoionization detector or an organic vapor analyzer (flame ionization detector), and record the measurements.

2. Lower an interface probe into the well to determine the existence of any immiscible layer(s), LNAPL and/or DNAPL, and record the measurements.
3. Confirm the presence or absence of an immiscible phase by slowly lowering a clear bailer to the appropriate depth, then visually observing the results after sample recovery.
4. In rare instances, such as when very viscous product is present, it may be necessary to utilize hydrocarbon- and water-sensitive pastes for measurement of LNAPL thickness. This is accomplished by smearing adjacent, thin layers of both hydrocarbon- and water-sensitive pastes along a steel measuring tape and inserting the tape into the well. An engineering tape showing tenths and hundredths of feet is required. Record depth to water, as shown by the mark on the water-sensitive paste, and depth to product, as shown by the mark on the product-sensitive paste. In wells where the approximate depth to water and product thickness are not known, it is best to apply both pastes to the tape over a fairly long interval (5 feet or more). Under these conditions, measurements are obtained by trial and error, and may require several insertions and retrievals of the tape before the paste-covered interval of the tape encounters product and water. In wells where approximate depths of air-product and product-water interfaces are known, pastes may be applied over shorter intervals. Water depth measurements should not be used in preparation of water-table contour maps until they are corrected for depression by the product.

If the well contains an immiscible phase, it may be desirable to sample this phase separately. Sections 5.3.5.1 and 5.3.5.2 present immiscible phase sampling procedures. It may not be meaningful to conduct water sample analysis of water obtained from a well containing LNAPLs or DNAPLs. Consult the CTO Manager and QA Manager or Technical Director if this situation is encountered.

5.3.4 Purging Equipment and Use

The water present in a well prior to sampling may not be representative of *in situ* groundwater quality and shall be removed prior to sampling. Handle all groundwater removed from potentially contaminated wells in accordance with the investigation-derived waste (IDW) handling procedures in Procedure I-A-6, *Investigation-Derived Waste Management.*

Purging shall be accomplished by removing groundwater from the well at low flow rates using a pump. According to the U.S. Environmental Protection Agency (EPA) (EPA 1996), the rate at which groundwater is removed from the well during purging ideally should be less than 0.2 to 0.3 liters/min. The EPA further states that wells should be purged at rates below those used to develop the well to prevent further development of the well, to prevent damage to the well, and to avoid disturbing accumulated corrosion or reaction products in the well. The EPA also indicates that wells should be purged at or below their recovery rate so that migration of water in the formation above the well screen does not occur.

Realistically, the purge rate should be low enough that substantial drawdown in the well does not occur during purging. The goal is minimal drawdown (less than 0.1 meter) during purging (EPA 1996). The amount of drawdown during purging should be recorded at the same time the other water parameters are measured. Also, a low purge rate will reduce the possibility of stripping volatile organic compounds (VOCs) from the water, and will reduce the likelihood of mobilizing colloids in the subsurface that are immobile under natural flow conditions.

The sampler shall ensure that purging does not cause formation water to cascade down the sides of the well screen. Wells shall not be purged to dryness if recharge causes the formation water to cascade down the sides of the screen, as this will cause an accelerated loss of volatiles. This problem should be anticipated. Water shall be purged from the well at a rate that does not cause recharge water to be excessively agitated unless an extremely slow recharging well is encountered where complete evacuation is unavoidable.

In high yield wells (wells that exhibit 80 percent recovery in less than 2 hours), purging shall be conducted at relatively low flow rates and shall remove water from the entire screened interval of the well to ensure that fresh water from the formation is present throughout the entire saturated interval. In general, place the intake of the purge pump 2 to 3 feet below the air-water interface within the well to allow purging and at the same time minimize disturbance/overdevelopment of the screened interval in the well. During the well purging procedure, collect water level and/or product level measurements to assess the hydraulic effects of purging. Sample the well when it recovers sufficiently to provide enough water for the analytical parameters specified.

Low yield wells (those that exhibit less than 80 percent recovery in less than 2 hours) require one borehole volume of water to be removed. Allow the well to recover sufficiently to provide enough water for the specified analytical parameters, and then sample it.

Evaluate water samples on a regular basis (approximately every 5 minutes) during well evacuation and analyze them in the field preferably using a multi-parameter meter and flow-through cell for temperature, pH (indicates the hydrogen ion concentration – acidity or basicity), specific conductivity, dissolved oxygen (DO), oxidation reduction potential (ORP), turbidity, salinity, and total dissolved solids (TDS). Take at least five readings during the purging process. These parameters are measured to demonstrate that the natural character of the formation water has been pumped into the well. Purging shall be considered complete when three consecutive sets of field parameter measurements stabilize within approximately 10 percent (EPA 2006). However, suggested ranges are ±0.2 degrees Celsius for temperature, ±0.1 standard units for pH, ±3 percent for specific conductance, ±10 percent for DO, and ±10 millivolts for redox potential (ASTM 2001). This criterion may not be applicable to temperature if a submersible pump is used during purging due to the heating of the water by the pump motor. Enter all information obtained during the purging and sampling process including drawdown, into a groundwater sampling log (Figure I-C-3-1). Complete all blanks on this field log during sampling.

In cases where an LNAPL has been detected in the monitoring well, insert a stilling tube of a minimum diameter of 2 inches into the well prior to well purging. The stilling tube shall be composed of a material that meets the performance guidelines for sampling devices. Insert the stilling tube into the well to a depth that allows groundwater from the screened interval to be purged and sampled, but that is below the upper portion of the screened interval where the LNAPL is entering the well screen. The goal is to sample the aqueous phase (groundwater) while preventing the LNAPL from entering the sampling device. To achieve this goal, insert the stilling tube into the well in a manner that prevents the LNAPL from entering the stilling tube. However, sampling groundwater beneath a NAPL layer is not generally recommended due to the fact that the interval with residual NAPL saturation is often unknown and the NAPL can be mobilized into the well from intervals below the water table.

One method of doing this is to cover the end of the stilling tube with a membrane or material that will be ruptured by the weight of the pump. A piece of aluminum foil can be placed over the end of the stilling tube. Slowly lower the stilling tube into the well to the appropriate depth and then attach it firmly to the top of the well casing. When the pump is inserted, the weight of the pump breaks the foil covering the end of the tube, and the well can be purged and sampled from below the LNAPL layer. Firmly fasten the membrane or material that is used to cover the end of the stilling tube so that it remains attached to the stilling tube when ruptured. Moreover, the membrane or material must retain its integrity after it is ruptured. Pieces of the membrane or material must not fall off of the stilling tube into the well. Although aluminum foil is mentioned in this discussion as an example of a material that can be used to cover the end of the tube, a more chemically inert material may be required, based on the site-specific situation. Thoroughly decontaminate stilling tubes prior to each use. Collect groundwater removed during purging, and store it on site until its disposition is determined based upon laboratory analytical results. Storage shall be in secured containers, such as U.S. Department of Transportation-approved drums. Label containers of purge water with the standard NAVFAC Pacific ER Program IDW label.

The following paragraphs list available purging equipment and methods for their use.

5.3.4.1 BAILERS AND PUMPS

Submersible Pump: A stainless steel submersible pump may be utilized for purging both shallow and deep wells prior to sampling groundwater for volatile, semivolatile, and non-volatile constituents. For wells over 200 feet deep, the submersible pump is one of the few technologies available to feasibly accomplish purging under any yield conditions. For shallow wells with low yields, submersible pumps are generally inappropriate due to over stressing of the wells (<1 gallon per minute), which causes increased aeration of the water within the well.

Steam clean or otherwise decontaminate the pump and discharge tubing prior to the placing the pump in the well. The submersible pump shall be equipped with an anti-backflow check valve to keep water from flowing back down the drop pipe into the well. Place the pump intake approximately 2 to 3 feet below the air-water interface within the well and maintain it in that position during purging. Additionally, when pulling the pump out of the well subsequent to purging, take care to avoid dumping water within the drop pipe and pump stages back into the well.

Bladder Pump: A stainless steel and/or Teflon bladder pump can be utilized for purging and sampling wells up to 200 feet in depth for volatile, semivolatile, and non-volatile constituents. Additionally, the bladder pump can be used for purging and obtaining groundwater samples overlain by a LNAPL layer as long as care is taken not to draw the product layer into the bladder pump. Use of the bladder pump is most effective in low to moderate yield wells.

Either a battery powered compressor, compressed dry nitrogen, or compressed dry air, depending upon availability, can operate the bladder pump. The driving gas utilized must be dry to avoid damage to the bladder pump control box. Decontaminate the bladder pump prior to use. Once purging is complete, collect the samples directly from the bladder pump.

Centrifugal or Diaphragm Pump: A centrifugal, or diaphragm, pump may be used to purge a well if the water level is within 20 feet of ground surface. A new, or properly decontaminated, hose is lowered into the well and water withdrawn at a rate that does not cause excessive well drawdown.

GROUNDWATER SAMPLING LOG

WELL NO. _____ LOCATION: _____ PROJECT NO. _____

DATE: TIME: _____ CLIMATIC CONDITIONS: _____

TIDAL CONDITIONS: Rising ☐ Falling ☐ HIGH TIDE: LOW TIDE: CURRENT TIDE:

STATIC WATER LEVEL (FT.) and TIME: _____ TOTAL DEPTH (FT.): _____

WELL PURGING: LENGTH OF SATURATED ZONE: _____ LINEAR FT.

a VOLUME OF WATER TO BE EVACUATED: _____ GALS. (Gals/Linear ft. X linear feet of saturation X 3-casing volumes)

METHOD OF REMOVAL: _____ PUMPING RATE: _____ mL/min

WELL PURGE DATA:

DATE/ TIME	DTW	GALLONS REMOVED	TDS (g/L)	pH	SP. COND. (mS/cm)	D.O. (mg/L)	TURB. (NTU)	TEMP. (°C)	ORP (mV)	SAL (ppt)

SAMPLE WITHDRAWAL METHOD: _____

APPEARANCE OF SAMPLE: COLOR: _____

SEDIMENT: _____

OTHER: _____

LABORATORY ANALYSIS PARAMETERS AND PRESERVATIVES _____

NUMBER AND TYPES OF SAMPLE CONTAINERS USED: _____

SAMPLE IDENTIFICATION NUMBER(S) _____

DECONTAMINATION PROCEDURES: _____

NOTES: _____

SAMPLED BY: _____

SAMPLES DELIVERED TO: _____ TRANSPORTER: _____

DATE: _____ TIME: _____

CAPACITY OF CASING (GALLONS/LINEAR FOOT)

2"-0.16•4"-0.65•6"-1.47•8"-2.61•10"-4.08•12"-5.87

Figure I-C-3-1: Groundwater Sampling Log

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Place the hose bottom approximately 2 to 3 feet below the air-water interface and maintain it in that position during purging.

Air Lift Pump: Airlift pumps are not appropriate for purging or sampling.

Bailer: Avoid using a bailer to purge a well because it can result in aeration of the water in the well and possibly cause excessive purge rates. If a bailer must be used, decontaminate the bailer, bailer wire, and reel as described in Section 5.3.2 prior to its use. Teflon-coated cable mounted on a reel is recommended for lowering the bailer in and out of the well.

Lower the bailer below the water level of the well with as little disturbance of the water as possible to minimize aeration of the water in the well. One way to gauge the depth of water on the reel is to mark the depth to water on the bailer wire with a stainless steel clip. In this manner, less time is spent trying to identify the water level in the well. The QA Manager or Technical Director shall approve use of bailers for purging monitoring wells in advance.

5.3.5 Monitoring Well Sampling Methodologies

5.3.5.1 Sampling Light, Non-Aqueous Phase Liquids (LNAPL)

Collect LNAPL, if present, prior to any purging activities. The sampling device shall generally consist of a dedicated or disposable bailer equipped with a bottom-discharging device. Lower the bailer slowly until contact is made with the surface of the LNAPL, and to a depth less than that of the immiscible fluid/water interface depth as determined by measurement with the interface probe. Allow the bailer to fill with the LNAPL and retrieve it.

When sampling LNAPLs, never drop bailers into a well, and always remove them from the well in a manner that causes as little agitation of the sample as possible. For example, the bailer should not be removed in a jerky fashion or be allowed to continually bang against the well casing as it is raised. When using bailers to collect LNAPL samples for inorganic analyses, the bailer shall be composed of fluorocarbon resin. Bailers used to collect LNAPL samples for organic analyses shall be constructed of stainless steel. The cable used to raise and lower the bailer shall be composed of an inert material (e.g., stainless steel) or coated with an inert material (e.g., Teflon).

5.3.5.2 Sampling Dense, Non-Aqueous Phase Liquids (DNAPL)

Collect DNAPL prior to any purging activities. The best method for collecting DNAPL is to use a double-check valve, stainless steel bailer, or a Kemmerer (discrete interval) sampler. The sample shall be collected by slow, controlled lowering of the bailer to the bottom of the well, activation of the closing device, and retrieval.

5.3.5.3 Groundwater Sampling Methodology

The well shall be sampled when groundwater within it is representative of aquifer conditions and after it has recovered sufficiently to provide enough volume for the groundwater sampling parameters. A period of no more than 2 hours shall elapse between purging and sampling to prevent groundwater interaction with the casing and atmosphere. This may not be possible with a slowly recharging well. Measure and record the water level prior to sampling to demonstrate the degree of recovery of the well. Sampling equipment (e.g., especially bailers) shall never be dropped into the well, as this could cause aeration of the water upon impact. Additionally, the sampling methodology utilized shall allow for the collection of a groundwater sample in as undisturbed a condition as

possible, minimizing the potential for volatilization or aeration. This includes minimizing agitation and aeration during transfer to sample containers.

Sampling equipment shall be constructed of inert material. Equipment with neoprene fittings, polyvinyl chloride bailers, tygon tubing, silicon rubber bladders, neoprene impellers, polyethylene, and viton is not acceptable. If bailers are used, an inert cable/chain (e.g., fluorocarbon resin-coated wire or single strand stainless steel wire) shall be used to raise and lower the bailer. Generally, bladder and submersible pumps are acceptable sampling devices for all analytical parameters. Dedicated equipment is highly recommended for all sampling programs. The following text describes sampling methods utilizing submersible pumps, bladder pumps, and bailers.

Submersible Pumps: When operated under low-flow rate conditions (100 to 300 milliliters [mL]/minute or less), submersible pumps are as effective as bladder pumps in acquiring samples for volatile organic analysis as well as other analytes. The submersible pump must be specifically designed for groundwater sampling (i.e., pump composed of stainless steel and Teflon, sample discharge lines composed of Teflon) and must have a controller mechanism allowing the required low flow rate. Adjust the pump rate so that flow is continuous and does not pulsate to avoid aeration and agitation within the sample discharge lines. Run the pump for several minutes at the low flow rate used for sampling to ensure that the groundwater in the lines was obtained at the low flow rate. Higher pumping rates than 100 to 300 mL/minute may be used when collecting samples to be analyzed for non-volatile constituents, if significant drawdown does not occur.

Bladder Pumps: A gas-operated Teflon or stainless steel bladder pump with adjustable flow control and equipped with Teflon-lined tubing can be effectively utilized to collect a groundwater sample and is considered to be the best overall device for sampling inorganic and organic constituents. Operate positive gas displacement bladder pumps in a continuous manner so that they minimize discharge pulsation that can aerate samples in the return tube or upon discharge. If a bladder pump is utilized for the well purging process, the same bladder pump can also be utilized for sample collection after purging is complete.

Most models of bladder pumps can be operated with a battery powered compressor and control box. The compressor can be powered with either a rechargeable battery pack (provided with the compressor), by running directly off of a vehicle battery (via alligator clips), or by plugging into the vehicle's direct current connector (cigarette lighter receptacle). When using a vehicle to power a compressor, several precautions should be taken. First, position the vehicle downwind of the well. Second, ensure the purge water exiting the well is collected into a drum or bucket. Finally, connect the compression hose from the well cap to the control box. Do not connect the compression hose from the compressor to the control box until after the engine has been started.

When all precautions are completed and the engine has been started, connect the compression hose to the control box. Slowly adjust the control knobs so as to discharge water at a flow rate (purge rate) that minimizes drawdown in the well, usually around 100 to 300 mL/minute. The compressor should not be set as to discharge the water as hard as possible. The optimal setting is one that produces the required purge rate per minute (not per purge cycle) while maintaining a minimal drawdown.

Prior to sampling volatiles constituents, turn off the vehicle engine, and obtain a flow rate of 100 mL/minute so as not to cause fluctuation in pH, pH-sensitive analytes, the loss of volatile constituents, or draw down of the groundwater table. If necessary (when sampling wells that require

a large sample volume) the vehicle engine may be turned back on after sampling volatile constituents. Higher flow rates (100 to 300 mL/minute) can be used once the samples for the analysis of volatile components have been collected, but should not allow for increased draw down in the well. At no time shall the sample flow rate exceed the flow rate used while purging. Preserve the natural conditions of the groundwater, as defined by pH, DO, specific conductivity, and reduction/oxidation (redox).

For those samples requiring filtration, it is recommended to use in-line high capacity filters after all nonfiltered samples have been collected.

Bailers: A single- or double-check valve Teflon or stainless steel bailer equipped with a bottom discharging device can be utilized to collect groundwater samples. Bailers have a number of disadvantages, however, including a tendency to alter the chemistry of groundwater samples due to degassing, volatilization, and aeration; the possibility of creating high groundwater entrance velocities; differences in operator techniques resulting in variable samples; and difficulty in determining where in the water column the sample was collected. Therefore, use bailers for groundwater sampling only when other types of sampling devices cannot be utilized for technical or logistical reasons. The QA Manager or Technical Director must approve the use of bailers for groundwater sampling in advance.

Thoroughly decontaminate the bailer before being lowering it into the well if it is not a disposable bailer sealed in plastic. Collect two to three rinse samples and discharge them prior to acquisition of the actual sample. Each time the bailer is lowered to the water table, lower it in such a way as to minimize disturbance and aeration of the water column within the well.

The preferred alternative when using bailers for sampling is to use disposable Teflon bailers equipped with bottom-discharging devices. Use of disposable bailers reduces decontamination time and limits the potential for cross-contamination.

Passive Sampling: Passive samplers include passive diffusion bags, HydraSleeve, Snap Sampler, Gore Sorbers, and rigid porous polyethylene samplers. Passive samplers generate minimal waste and purge water, if any. Passive samplers depend on ambient equilibrium with formation water. These are relatively inexpensive, simple to deploy and work well for low-yield wells. However, passive samplers have volume and or analyte limitations and may require consideration of contaminant stratification. Passive samplers should be handled in accordance with the manufacturer's instructions, Army guidance (USACE 2002), or ITRC guidance (ITRC 2007).

5.3.6 Sample Handling and Preservation

Many of the chemical constituents and physiochemical parameters to be measured or evaluated during groundwater monitoring programs are chemically unstable; therefore, preserve samples. The EPA document entitled, *Test Methods for Evaluating Solid Waste – Physical/Chemical Methods, SW-846* (EPA 2007), includes a discussion of appropriate sample preservation procedures. In addition, SW-846 specifies the sample containers to use for each constituent or common set of parameters. In general, check with specific laboratory requirements prior to obtaining field samples. In many cases, the laboratory will supply the necessary sample bottles and required preservatives. In some cases, the field team may add preservatives in the field. Sample containers should be labeled in accordance with Procedure III-E, *Record Keeping, Sample Labeling, and Chain of Custody.*

Improper sample handling may alter the analytical results of the sample. Therefore, transfer samples in the field from the sampling equipment directly into the container that has been prepared specifically for that analysis or set of compatible parameters as described in the CTO-specific work plan. It is not an acceptable practice for samples to be composited in a common container in the field and then split in the laboratory, or poured first into a wide mouth container and then transferred into smaller containers.

Collect groundwater samples and place them in their proper containers in the order of decreasing volatility and increasing stability. A preferred collection order for some common groundwater parameters is:

1. VOCs and total organic halogens (TOX)
2. Dissolved gases, total organic carbon (TOC), total fuel hydrocarbons
3. Semivolatile organics, pesticides
4. Total metals, general minerals (unfiltered)
5. Dissolved metals, general minerals (filtered)
6. Phenols
7. Cyanide
8. Sulfate and chloride
9. Turbidity
10. Nitrate and ammonia
11. Radionuclides

When sampling for VOCs, collect water samples in vials or containers specifically designed to prevent loss of VOCs from the sample. An analytical laboratory shall provide these vials, preferably by the laboratory that will perform the analysis. Collect groundwater from the sampling device in vials by allowing the groundwater to slowly flow along the sides of the vial. Sampling equipment shall not touch the interior of the vial. Fill the vial above the top of the vial to form a positive meniscus with no overflow. No headspace shall be present in the sample container once the container has been capped. This can be checked by inverting the bottle once the sample is collected and tapping the side of the vial to dislodge air bubbles. Sometimes it is not possible to collect a sample without air bubbles, particularly water that is aerated. In these cases, the investigator shall note the problem to account for possible error. Cooling samples may also produce headspace, but this will typically disappear once the sample is warmed prior to analysis. In addition, if the samples are shipped by air, air bubbles form most of the time. Field logs and laboratory analysis reports shall note any headspace in the sample container(s) at the time of receipt by the laboratory, as well as at the time the sample was first transferred to the sample container at the wellhead.

5.3.6.1 SPECIAL HANDLING CONSIDERATIONS

Samples requiring analysis for organics shall not be filtered. Samples shall not be transferred from one container to another because this could cause aeration or a loss of organic material onto the walls of the container. TOX and TOC samples shall be handled and analyzed in the same manner as VOC samples.

Obtain groundwater samples to be analyzed for metals sequentially. One sample shall be obtained directly from the pump and be unfiltered. The second sample shall be filtered through a 0.45-micron membrane in-line filter. Both filtered and unfiltered samples shall be transferred to a container, preserved with nitric acid to a pH less than 2, and analyzed for dissolved metals. Remember to include a filter blank for each lot of filters used and always record the lot number of the filters. In addition, allow at least 500 mL of effluent to flow through the filter prior to sampling. Any difference in concentration between the total and dissolved fractions may be attributed to the original metallic ion content of the particles and adsorption of ions onto the particles.

5.3.6.2 FIELD SAMPLING PRESERVATION

Preserve samples immediately upon collection. Ideally, sampling containers will be pre-preserved with a known concentration and volume of preservative. For example, metals require storage in aqueous media at pH of 2 or less. Typically, 0.5 mL of 1:1 nitric acid added to 500 mL of groundwater will produce a pH less than 2. Certain matrices that have alkaline pH (greater than 7) may require more preservative than is typically required. An early assessment of preservation techniques, such as the use of pH strips after initial preservation, may therefore be appropriate. The introduction of preservatives will dilute samples, and may require normalization of results. Guidance for the preservation of environmental samples can be found in the EPA *Handbook for Sampling and Sample Preservation of Water and Wastewater* (EPA 1982). Additional guidance can be found in other EPA documents (EPA 1992, 1996).

5.3.6.3 FIELD SAMPLING LOG

A groundwater sampling log (Figure I-C-3-1) shall document the following:

- Identification of well
- Well depth
- Static water level depth and measurement technique
- Presence of immiscible layers and detection method
- Well yield
- Purge volume and pumping rate
- Time that the well was purged
- Collection method for immiscible layers
- Sample identification numbers
- Well evacuation procedure/equipment
- Sample withdrawal procedure/equipment
- Date and time of collection
- Well sampling sequence
- Types of sample containers used and sample identification numbers
- Preservative(s) used
- Parameters requested for analysis

- Field analysis data
- Sample distribution and transporter
- Field observations on sampling event
- Name of collector
- Climatic conditions including air temperature

6. Records

Document information collected during groundwater sampling on the groundwater sampling log form in indelible ink (Figure I-C-3-1). Send copies of this information to the CTO Manager and to the project files.

7. Health and Safety

Field personnel shall perform work in accordance with the current (or as contractually obligated) United States Army Corps of Engineers Safety and Health Requirements Manual EM-385-1-1 (USACE 2008) and site-specific health and safety plan.

8. References

ASTM International (ASTM). 2001. *Standard Guide for Sampling Ground-Water Monitoring Wells*. D4448). Reapproved in 2013). West Conshohocken, PA.

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual*. OPNAV Instruction 5090.1D. 10 January.

Environmental Protection Agency, United States (EPA). 1982. *Handbook for Sampling and Sample Preservation of Water and Wastewater*. EPA-600/4-82-029. Cincinnati: EPA Office of Research and Development, Environmental Monitoring and Support Laboratory.

———. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

———. 1996. *Ground Water Issue: Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*. EPA/540/S-95/504. Office of Solid Waste and Emergency Response. April.

———. 2006. *Systematic Planning: A Case Study for Hazardous Waste Site Investigations. EPA WA/CS-1*. EPA/240/B-06/004. Office of Environmental Information. March.

———. 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*. 3rd ed., Revision 6. Office of Solid Waste. November. On-line updates at: http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm.

Interstate Technology and Regulatory Council (ITRC). 2007. *Protocol for Use of Five Passive Samplers to Sample for a Variety of Contaminants in Groundwater.* February.

United States Army Corps of Engineers (USACE). 2002. *Study of Five Discrete Interval-Type Groundwater Sampling Devices*. Cold Regions Research and Engineering Laboratory. Hanover, NH. August.

———. 2008. *Consolidated Safety and Health Requirements Manual.* EM-385-1-1. Includes Changes 1–7. 13 July 2012.

Procedure I-A-6, *Investigation-Derived Waste Management.*

Procedure I-F, *Equipment Decontamination.*

Procedure III-B, *Field QC Samples (Water, Soil).*

Procedure III-E, *Record Keeping, Sample Labeling, and Chain of Custody.*

9. Attachments

None.

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Equipment Decontamination

1. Purpose

This standard operating procedure describes methods of equipment decontamination for use during site activities by United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific personnel.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

None.

4. Responsibilities

The prime contractor CTO Manager is responsible for identifying instances of non-compliance with this procedure and ensuring that decontamination activities comply with this procedure. The CTO Manager is responsible for ensuring that all personnel involved in equipment decontamination have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

The prime contractor QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

The Field Manager is responsible for field oversight to ensure that all project field staff follow these procedures.

Field personnel are responsible for the implementation of this procedure.

5. Procedures

Decontamination of equipment used in sampling of various media, groundwater monitoring, and well drilling and development is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program requires consideration of the following factors:

- The location where the decontamination procedures will be conducted
- The types of equipment requiring decontamination
- The frequency of equipment decontamination
- The cleaning technique and types of cleaning solutions appropriate for the contaminants of concern
- The method for containing the residual contaminants and wash water from the decontamination process
- The use of a quality control measure to determine the effectiveness of the decontamination procedure

The following subsection describes standards for decontamination, including the frequency of decontamination, cleaning solutions and techniques, containment of residual contaminants and cleaning solutions, and effectiveness.

5.1 DECONTAMINATION AREA

Select an appropriate location for the decontamination area at a site based on the ability to control access to the area, the ability to control residual material removed from equipment, the need to store clean equipment, and the ability to restrict access to the area being investigated. Locate the decontamination area an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

It is the responsibility of the site safety and health officer (SSHO) to set up the site zones (i.e., exclusion, transition, and clean) and decontamination areas. Generally, the decontamination area is located within the transition zone, upwind of intrusive activities, and serves as the washing area for both personnel and equipment to minimize the spread of contamination into the clean zone. For equipment, a series of buckets are set up on a visqueen-lined bermed area. Separate spray bottles containing laboratory-grade isopropyl alcohol (or alternative cleaning solvent as described in the CTO work plan [WP]) and distilled water are used for final rinsing of equipment. Depending on the nature of the hazards and the site location, decontamination of heavy equipment, such as augers, pump drop pipe, and vehicles, may be accomplished using a variety of techniques.

5.2 TYPES OF EQUIPMENT

Drilling equipment that must be decontaminated includes drill bits, auger sections, drill-string tools, drill rods, split barrel samplers, tremie pipes, clamps, hand tools, and steel cable. Decontamination of monitoring well development and groundwater sampling equipment includes submersible pumps, bailers, interface probes, water level meters, bladder pumps, airlift pumps, peristaltic pumps, and lysimeters. Other sampling equipment that requires decontamination includes, but is not limited to, hand trowels, hand augers, slide hammer samplers, shovels, stainless-steel spoons and bowls, soil sample liners and caps, wipe sampling templates, composite liquid waste samplers, and dippers. However, equipment that is shipped pre-packaged from the vendor should not have to be decontaminated prior to first use. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and shall be properly disposed of after one use.

5.3 FREQUENCY OF EQUIPMENT DECONTAMINATION

Decontaminate down-hole drilling equipment and equipment used in monitoring well development and purging prior to initial use and between each borehole or well. Down-hole drilling equipment, however, may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, decontaminate the drilling tools prior to drilling deeper. Initiate groundwater sampling by sampling groundwater from the monitoring well where the least contamination is suspected. Decontaminate groundwater, surface water, and soil sampling devices prior to initial use and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

5.4 CLEANING SOLUTIONS AND TECHNIQUES

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment, such as drill bits, augers, drill string, and pump drop-pipe, is steam cleaning. To steam clean, use a portable, high-pressure steam cleaner equipped with a pressure hose and fittings. For this method, thoroughly steam wash equipment, and rinse it with potable tap water to remove particulates and contaminants.

Where appropriate, disposable materials are recommended. A rinse decontamination procedure is acceptable for equipment, such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a non-phosphate detergent (alconox, liquinox, or other suitable detergent) and potable water solution; (2) rinse in a bath with potable water; (3) spray with laboratory-grade isopropyl alcohol; (4) rinse in a bath with deionized or distilled water; and (5) spray with deionized or distilled water. If possible, disassemble equipment prior to cleaning. Add a second wash at the beginning of the process if equipment is very soiled.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. Decontaminate these pumps by washing and rinsing the outside surfaces using the procedure described for small equipment or by steam cleaning. Decontaminate the internal surfaces by recirculating fluids through the pump while it is operating. This recirculation may be done using a relatively long (typically 4 feet) large-diameter pipe (4-inch or greater) equipped with a bottom cap. Fill the pipe with the decontamination fluids, place the pump within the capped pipe, and operate the pump while recirculating the fluids back into the pipe. The decontamination sequence shall include: (1) detergent and potable water; (2) potable water rinse; (3) potable water rinse; and (4) deionized water rinse. Change the decontamination fluids after each decontamination cycle.

Solvents other than isopropyl alcohol may be used, depending upon the contaminants involved. For example, if polychlorinated biphenyls or chlorinated pesticides are contaminants of concern, hexane may be used as the decontamination solvent. However, if samples are also to be analyzed for volatile organics, hexane shall not be used. In addition, some decontamination solvents have health effects that must be considered. Decontamination water shall consist of distilled or deionized water. Steam-distilled water shall not be used in the decontamination process as this type of water usually contains elevated concentrations of metals. Decontamination solvents to be used during field activities will be specified in CTO WP and site-specific health and safety plan.

Rinse equipment used for measuring field parameters, such as pH, temperature, specific conductivity, and turbidity with deionized or distilled water after each measurement. Also wash new, unused soil sample liners and caps with a fresh detergent solution and rinse them with potable water followed by distilled or deionized water to remove any dirt or cutting oils that might be on them prior to use.

5.5 Containment of Residual Contaminants and Cleaning Solutions

A decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water.

When contaminated material and cleaning fluids must be contained from heavy equipment, such as drilling rigs and support vehicles, the area must be properly floored, preferably with a concrete pad that slopes toward a sump pit. If a concrete pad is impractical, planking can be used to construct solid flooring that is then covered by a nonporous surface and sloped toward a collection sump. If the decontamination area lacks a collection sump, use plastic sheeting and blocks or other objects to create a bermed area for collection of equipment decontamination water. Situate items, such as auger flights, which can be placed on metal stands or other similar equipment, on this equipment during decontamination to prevent contact with fluids generated by previous equipment decontamination. Store clean equipment in a separate location to prevent recontamination. Collect decontamination fluids contained within the bermed area and store them in secured containers as described below.

Use wash buckets or tubs to catch fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices. Collect the decontamination fluids and store them on site in secured containers, such as U.S. Department of Transportation-approved drums, until their disposition is determined by laboratory analytical results. Label containers in accordance with Procedure I-A-6, *Investigation-Derived Waste Management*.

5.6 Effectiveness of Decontamination Procedures

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment blank samples or wipe testing. Equipment blanks consist of analyte-free water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a cloth over the surface of the equipment after cleaning. Procedure III-B, *Field QC Samples (Water, Soil)* provides further descriptions of these samples and their required frequency of collection. These quality control measures provide "after-the fact" information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

6. Records

Describe the decontamination process in the field logbook.

7. Health and Safety

Field Personnel shall perform work in accordance with the current (or as contractually obligated) United States Army Corps of Engineers Safety and Health Requirements Manual EM-385-1-1 (USACE 2008) and site-specific health and safety plan.

8. References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual*. OPNAV Instruction 5090.1D. 10 January.

United States Army Corps of Engineers (USACE). 2008. *Consolidated Safety and Health Requirements Manual.* EM-385-1-1. Includes Changes 1–7. 13 July 2012.

Procedure I-A-6, *Investigation-Derived Waste Management.*

Procedure III-B, *Field QC Samples (Water, Soil).*

9. Attachments

None.

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Data Validation

1. Purpose

This procedure describes the presentation format and information provided in the data validation reports under the United States (U.S.) Navy Environmental Restoration (ER) Program for Naval Facilities Engineering Command (NAVFAC), Pacific. The objective of data validation is to provide data of known quality to the end user. This procedure also establishes the method by which a Contract Task Order (CTO) Manager selects and confirms the content of data validation reports and is consistent with protocol in the *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories* (DoD QSM) (DoD 2013).

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012) and 2B (2005b) as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA manager) shall also concur with any deviations.

3. Definitions

Acronyms and abbreviations used in all data validation procedures and reports are defined in Attachment II-A-1. Commonly used terms are defined in Attachment II-A-2.

4. Responsibilities

The CTO Manager, the QA Manager or Technical Director, and the CTO QA Coordinator are responsible for ensuring that this procedure is implemented by data validation personnel.

Data validation personnel are responsible for implementing this procedure for all data validation reports.

5. Procedure

5.1 INTRODUCTION

This procedure addresses the validation of data obtained under the NAVFAC Pacific ER Program using primarily U.S. Environmental Protection Agency (EPA) Solid Waste (SW)-846 methods (EPA 2007). Based on the data validation requirements identified in the CTO project planning documents, the analytical data may undergo "Level B," "Level C," or "Level D" data validation or

some combination of these validation levels. This procedure establishes the required format and content of the various validation reports.

5.1.1 Confirmation of Data Validation Reports

Prior to shipment of all completed data validation reports to the CTO Manager, a single draft report for one sample delivery group (SDG) should be submitted. The CTO Manager shall review the draft report to confirm that the report contains the requested information, and respond to the Data Validation Project Manager in a timely manner. Once the requested contents are confirmed, the complete data validation packages should be delivered to the CTO Manager.

5.2 CONTENT AND FORMAT OF THE DATA VALIDATION REPORT

The data validation report will consist of the following four major components:

1. Cover letter
2. Data validation reference package comprising:
   a. Cover page
   b. Acronyms and abbreviations list
   c. Data qualifier reference table
   d. Qualification code reference table
3. Individual data validation reports by SDG:
   e. Cover page
   f. Introduction
   g. Data validation findings
   h. Appendix of laboratory reports with applied data qualifiers

A discussion of the contents and format of these components is provided in the following sections.

5.2.1 Cover Letter

The cover letter will contain the generation date of the cover letter, the address of the CTO office, the CTO number, and the CTO Manager's name or designee. The cover letter will list the specific reports being sent under that cover letter. A senior data reviewer must review the report and sign the cover letter to denote approval. Attachment II-A-3 is an example of the cover letter.

5.2.2 Data Validation Reference Package

One data validation reference package shall be provided per CTO and shall contain the reference information needed for interpretation of the individual data validation reports. The following sections shall be included:

5.2.2.1 COVER PAGE

The cover page shall indicate the CTO title and number to which the reference package applies.

5.2.2.2 ACRONYMS AND ABBREVIATIONS LIST

This list shall present all acronyms and abbreviations used in the individual data validation reports. Attachment II-A-1 is an example of the acronyms and abbreviations list.

5.2.2.3 DATA QUALIFIER REFERENCE TABLE

Data qualifiers are applied in cases where the data do not meet the required quality control (QC) criteria or where special consideration by the data user is required.

The data qualifier reference table lists the data qualifiers used in the validation of the analytical data. Attachment II-A-4 is an example of this table.

5.2.2.4 QUALIFICATION CODE REFERENCE TABLE

Qualification codes explain why data qualifiers have been applied and identify possible limitations of data use. Attachment II-A-5 provides the qualification codes used by the NAVFAC Pacific ER Program. Qualification codes are to be provided by data validation personnel on the annotated laboratory reports discussed in Section 5.2.3.4.

5.2.3 Individual Data Validation Reports by SDG

For all analyses, each SDG shall have a unique data validation report. The procedures used to generate the reports are discussed in the following sub-sections.

5.2.3.1 COVER PAGE

The cover page shall indicate the CTO title and number, analysis type, and the SDG(s), which the report addresses.

5.2.3.2 INTRODUCTION

This section will contain a brief description of the CTO information that is pertinent to data validation. This information includes the CTO title and number, CTO Manager, the sample matrices and analyses performed on the samples, the data validation level for the project, and a brief discussion of the methodologies used for data validation. This section will also contain a Sample Identification Table which lists the identification of each sample identification number cross referenced with its associated internal laboratory identification number and COC sample number. Each sample will be listed under every analytical method for which data was validated. Attachment II-A-6 is an example of the sample identification table.

5.2.3.3 DATA VALIDATION FINDINGS

This section shall present the data validation findings of the data reviewer for the CTO data package. The findings shall be determined on the basis of validation criteria established for each analytical method[1] in the DoD QSM (DoD 2013) or the CTO planning document and Procedure II-B through Procedure II-X. For all data validation levels, the data validation findings are divided into the following analytical categories:

- II-B GC/MS Volatile Organics by SW-846 Method 8260

[1] Other methods may be included with approval of the CTO and Data Validation Managers.

- II-C GC/MS Semivolatile Organics by SW-846 8270 (full scan and SIM)
- II-D HRGC/HRMS Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by SW-846 8290
- II-E Organochlorine Pesticides by SW-846 8081
- II-F Polychlorinated Biphenyls as Aroclors by SW-846 8082
- II-G Polychlorinated Biphenyls as Congeners by SW-846 8082
- II-H Total Petroleum Hydrocarbons by SW-846 8015
- II-I Chlorinated Herbicides by SW-846 8151
- II-J Organophosphorus Pesticides by SW-846 8141
- II-K Halogenated and Aromatic Volatiles by SW-846 8021
- II-L Phenols by SW-846 8041
- II-M Ethylene Dibromide/Dibromochloropropane by SW-846 8011
- II-N Polynuclear Aromatic Hydrocarbons by SW-846 8310
- II-O Explosives by SW-846 8330
- II-P Carbamate and Urea Pesticides by EPA Method 632
- II-Q Metals by EPA Method SW-846 6000/7000
- II-R Wet Chemistry Analyses
- II-S Data Quality Assessment Report
- II-T HRGC/HRMS Polychlorinated Biphenyls as Congeners by EPA Method 1668
- II-U Carbamate and Urea Pesticides by SW-846 8321
- II-V Perchlorate by SW-846 6850
- II-W GC/FID/ECD Volatile Organics and Fixed Gases in Soil Gas/Vapor by EPA Method TO-3 and ASTM D1946
- II-X GC/MS Volatile Organics and Fixed Gases in Soil Gas/Vapor by EPA Method TO-14, TO-15, and TO-17

GC/MS	gas chromatography/mass spectrometry
ECD	electron capture detector
FID	flame ionization detector
HRGC/HRMS	high resolution gas chromatograph/high resolution mass spectrometer
SIM	selective ion monitoring

Level C and Level D Data Validation

Data obtained using any analytical methods in the above categories will be validated in terms of meeting criteria for specific QA/QC factors such as holding times, instrument calibration, and blank analyses. A separate discussion of each QA/QC factor under each analytical method will be

presented in the CTO data validation report. The QA/QC factors used to validate data for Level C and Level D validation are presented below for each analytical category.

Volatile Organics by Gas Chromatography/Mass Spectrometry (GC/MS)

1. Sample management (sample preservation, handling, and transport, chain-of-custody, and holding times)
2. GC/MS instrument performance check
3. Calibration (initial calibration, initial calibration verification, and continuing calibration)
4. Method blanks
5. Blank spikes and laboratory control samples (LCSs)
6. Surrogate recovery
7. Matrix spike/matrix spike duplicate (MS/MSD)
8. Field QC samples (trip blanks, equipment blanks, field blanks, field duplicates, and field triplicates)
9. Internal standards performance
10. Target compound identification (Level D only*)
11. Compound quantitation and reporting limits (RLs) (Level D only*)
12. Tentatively identified compounds (Level D only*)
13. System performance (Level D only*)

Semivolatile Organics by Full Scan and SIM GC/MS

1. Sample management (sample preservation, handling, and transport; chain-of-custody; holding times)
2. GC/MS instrument performance check (full scan)
3. Calibration (initial calibration, initial calibration verification, and continuing calibration)
4. Method blanks
5. Blank spikes and LCSs
6. Surrogate recovery
7. MS/MSD
8. Field QC samples (equipment blanks, field blanks, and field duplicates)
9. Internal standards performance
10. Target Compound identification (Level D only*)
11. Compound quantitation and RLs (Level D only*)
12. Tentatively identified compounds (Level D only*)

13. System performance (Level D only*)

Dioxins/Dibenzofurans by HRGC/HRMS

1. Sample management (sample preservation, handling, and transport; chain-of-custody; holding times)
2. HRGC/HRMS instrument performance check
3. Calibration (initial calibration, initial calibration verification, and continuing calibration)
4. Method blanks
5. Blank spikes and LCSs
6. MS/MSD
7. Field QC samples (equipment blanks, field blanks, field duplicates, and field triplicates)
8. Internal standards performance
9. Target compound identification (Level D only*)
10. Compound quantitation and RLs (Level D only*)
11. System performance (Level D only*)

Organochlorine Pesticides by GC

1. Sample management (sample preservation, handling, and transport; chain-of-custody; holding times)
2. Pesticides instrument performance (retention time evaluation, 4,4'-DDT/Endrin breakdown evaluation)
3. Calibration (analytical sequence, initial calibration, initial calibration verification, continuing calibration)
4. Method blanks
5. Blank spikes and LCSs
6. Surrogate recovery
7. MS/MSD
8. Sample cleanup performance
9. Field QC samples (equipment blanks, field blanks, field duplicates, and field triplicates)
10. Target compound identification (Level D only*)
11. Compound quantitation and RLs (Level D only*)

Organic Analyses by GC (QA/QC factors may vary depending on analysis type)

1. Sample management (sample preservation, handling, and transport; chain-of-custody; holding times)

2. Instrument performance
3. Calibration (initial calibration, initial calibration verification and continuing calibration)
4. Method blanks
5. Blank spikes and LCS
6. Surrogate recovery
7. MS/MSD
8. Field QC samples (trip blanks [volatile organic compounds], equipment blanks, field blanks, field duplicates, and field triplicates)
9. Target compound identification (Level D only*)
10. Compound quantitation and RLs (Level D only*)

Organic Analyses by High-Performance Liquid Chromatography (QA/QC factors may vary depending on analysis type)

1. Sample management (sample preservation, handling, and transport; chain-of-custody; holding times)
2. Instrument performance
3. Calibration (initial calibration, initial calibration verification and continuing calibration)
4. Method blanks
5. Blank spikes and LCSs
6. Surrogate recovery
7. MS/MSD
8. Field QC samples (equipment blanks, field blanks, field duplicates, and field triplicates)
9. Target compound identification (Level D only*)
10. Compound quantitation and reporting limits (RLs) (Level D only*)

Organic Analyses by Liquid Chromatography–Mass Spectrometry (QA/QC factors may vary depending on analysis type)

1. Sample management (sample preservation, handling, and transport; chain-of-custody; holding times)
2. Instrument performance
3. Calibration (initial calibration, initial calibration verification, and continuing calibration)
4. Method blanks
5. Blank spikes and LCSs
6. MS/MSD
7. Field QC samples (equipment blanks, field blanks, field duplicates, and field triplicates)

8. Internal standards performance
9. Target compound identification (Level D only*)
10. Compound quantitation and RLs (Level D only*)

Metals

1. Sample management (sample preservation, handling, and transport; chain-of-custody; holding times)
2. Calibration (initial and continuing)
3. Blanks (Calibration blanks and Method [preparation] blanks)
4. Inductively coupled (argon) plasma (spectroscopy) (ICP) interference check sample
5. Blank spikes and LCSs
6. MS/MSD and Matrix duplicates
7. Furnace atomic absorption QC
8. Internal standards performance (MS methods only)
9. ICP serial dilution
10. Sample result verification (Level D only*)
11. Field QC samples (equipment blanks, field blanks, field duplicates, and field triplicates)

Inorganic Analyses by Wet Chemical Methods, (QA/QC factors may vary depending on analysis type)

1. Sample management (sample preservation, handling, and transport; chain-of-custody; and holding times)
2. Calibration (initial and continuing)
3. Method blanks
4. Blank spikes and LCSs
5. MS/MSD and Matrix duplicates
6. Sample result verification (Level D only*)
7. Field QC samples (equipment blanks, field blanks, field duplicates, and field triplicates)

* Sections applicable to Level D validation only will also appear in Level C validation reports with the notation "not applicable for Level C validation."

Level B Data Validation

Data obtained using any analytical methods in the Level B Validation analytical categories will be validated in terms of meeting criteria for specific QA/QC factors such as holding times, blank spike

analyses, and blank analyses. A separate discussion of each QA/QC factor under each analytical method will be presented in the CTO data validation report. The QA/QC factors used to validate data for QA/QC "Level B Validation" are presented below for each analytical category.

Organic Analyses

1. Sample management (sample preservation, handling, and transport; chain-of-custody; and holding times)
2. Method blanks
3. Blank spikes and laboratory control samples
4. Field QC samples (trip blanks (volatile organic compounds), equipment blanks, field blanks, field duplicates, and field triplicates)
5. Surrogate recovery
6. MS/MSD

Inorganic Analyses

1. Sample management (sample preservation, handling, and transport; chain-of-custody; and holding times)
2. Blanks (Calibration and Method blanks)
3. Blank spikes and LCSs
4. Field QC samples (equipment blanks, field blanks, field duplicates, and field triplicates)
5. MS/MSD and Laboratory Duplicates
6. ICP serial dilution

5.2.3.4 LABORATORY REPORTS

Annotated laboratory reports with the appropriate data qualifiers and qualification codes as specified in the NAVFAC Pacific ER Program data validation procedures will be submitted as an appendix to the data validation report. An example is provided as Attachment II-A-7. Records

Copies of all documents generated by data validation personnel will be stored for no less than 10 years. The original validated laboratory data shall be archived to the Federal Records Center at project completion.

6. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/swerffrr/pdf/-qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Environmental Protection Agency, United States (EPA). 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Final Update IV. Office of Solid Waste. On-line updates at: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

7. Attachments

Attachment II-A-1: Acronyms and Abbreviations

Attachment II-A-2: Definition of Terms

Attachment II-A-3: Sample Cover Letter

Attachment II-A-4: Data Qualifier Reference Table

Attachment II-A-5: Qualification Code Reference Table

Attachment II-A-6: Sample Identification Table

Attachment II-A-7: Example Annotated Laboratory Report Volatile Organics Analysis Data Sheet

Attachment II-A-1
Acronyms and Abbreviations

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ACRONYMS AND ABBREVIATIONS

Following is a list of acronyms and abbreviations that may be used in NAVFAC Pacific ER Program data validation reports and the data quality assessment reports.

%D	percent difference
%R	percent recovery
µg/kg	microgram per kilogram
µg/L	microgram per liter
4,4'-DDD	4,4'-dichlorodiphenyldichloroethane
4,4'-DDE	4,4'-dichlorodiphenyldichloroethylene
4,4'-DDT	4,4'-dichlorodiphenyltrichloroethane
AA	atomic absorption
ARRF	average relative response factor
BFB	bromofluorobenzene
BNA	base/neutral/acid
CCB	continuing calibration blank
CCC	calibration check compound
CCV	continuing calibration verification
CF	calibration factor
CLP	Contract Laboratory Program
COC	chain-of-custody
COD	chemical oxygen demand
CTO	contract task order
CVAA	cold vapor atomic absorption
DBCP	Dibromochloropropane
DCB	decachlorobiphenyl
DFTPP	decafluorotriphenylphosphine
DL	detection limit
DoD	Department of Defense
DOE	Department of Energy
DQAR	data quality assessment report
DUP	laboratory duplicate
DVP	data validation procedure
EB	equipment blank
EDB	ethylene dibromide
EDL	estimated detection limit
EICP	extracted ion current profile
EPA	Environmental Protection Agency, United States
FB	field blank
GC	gas chromatography
GC/ECD	gas chromatography/electron capture detector
GC/ELCD	gas chromatography/electrolytic conductivity detector (Hall detector)
GC/FPD	gas chromatography/flame photometric detector
GC/MS	gas chromatography/mass spectrometry

GC/PID	gas chromatography/photoionization detector
GFAA	graphite furnace atomic absorption
GPC	gel permeation chromatography
Hg	mercury
HPLC	high-performance liquid chromatography
HRGC/HRMS	high resolution gas chromatography/high resolution mass spectrometry
HT	holding time
ICB	initial calibration blank
ICP	inductively coupled plasma
ICS	interference check sample
ICV	initial calibration verification
IDL	instrument detection limit
IR	infrared spectroscopy
IRP	installation restoration program
IS	internal standards
LCS	laboratory control sample
LOD	limit of detection
LOQ	limit of quantitation
m/z	mass to charge ratio
MBAS	methyl blue active substance
mg/kg	milligram per kilogram
mg/L	milligram per liter
MS	matrix spike
MSA	method of standard addition
MSD	matrix spike duplicate
NFESC	Naval Facilities Engineering Services Center
ng/kg	nanogram per kilogram
OP	organophosphorus
PAH	polynuclear aromatic hydrocarbon
PARCC	precision, accuracy, representativeness, comparability, completeness
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzodioxin
PCDF	polychlorinated dibenzofuran
PE	performance evaluation
PEM	performance evaluation mixture
PFK	perfluorokerosene
pg/g	picogram per gram
pg/L	picogram per liter
PQO	project quality objective
QA	quality assurance
QAC	quality assurance coordinator
QAPP	quality assurance project plan
QC	quality control
QSM	quality system manual

r	correlation coefficient
r^2	coefficient of determination
RF	response factor
RIC	reconstructed ion chromatogram
RL	reporting limit
RPD	relative percent difference
RRF	relative response factor
RRT	relative retention time
RSD	relative standard deviation
RT	retention time
s/n	signal to noise ratio
SDG	sample delivery group
SICP	selected ion current profiles
SOP	standard operating procedure
SOW	statement of work
SPCC	system performance check compound
SRM	standard reference material
SVOC	semivolatile organic compound
TB	trip blank
TCDD	tetrachlorodibenzodioxin
TCX	tetrachloro-m-xylene
TDS	total dissolved solids
TIC	tentatively identified compound
TOC	total organic carbon
TOX	total organic halides
TPHE	total petroleum hydrocarbons as extractables
UV/VIS	ultraviolet/visible
VOA	volatile organic analysis
VOC	volatile organic compound
VTSR	validated time of sample receipt
WDM	window defining mixture

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Attachment II-A-2
Definition of Terms

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DEFINITION OF TERMS

Calibration Curve	–	A plot of response versus concentration of standards.
CCB	–	Continuing Calibration Blank – a deionized water sample run every 10 samples designed to detect any carryover contamination.
CCV	–	Continuing Calibration Verification – a standard run every 10 samples to test instrument performance.
EDL	–	Estimated Detection Limit – The sample specific EDL is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level.
Field Blank	–	Field blanks are intended to identify contaminants that may have been introduced in the field through source water.
Field Duplicate	–	A duplicate sample generated in the field, not in the laboratory.
Findings	–	Any out-of-control, unacceptable, or out of criteria event which may impact the quality of the data or require corrective action.
GPC	–	Gel Permeation Chromatography – A sample clean-up technique that separates compounds by size and molecular weight. Generally used to remove oily materials from sample extracts.
Holding Time	–	The time from sample collection to sample analysis.
ICB	–	Initial Calibration Blank – the first blank standard run to confirm the calibration curve.
ICV	–	Initial Calibration Verification – the first standard run to confirm the calibration curve.
Initial Calibration	–	The establishment of a calibration curve with the appropriate number of standards and concentration range. The calibration curve plots instrument response versus concentration of standards.
IR	–	Infrared Spectroscopy.
IS	–	Internal Standards – compounds added to every VOA and BNA standard, blank, matrix spike duplicate, and sample extract at a known concentration, prior to instrumental analysis. Internal standards are used as the basis for quantitation of the target compounds.
Laboratory Duplicate	–	A duplicate sample generated in the laboratory.
MDL	–	Method Detection Limit – minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.
MS	–	Matrix Spike – introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the extraction or digestion and measurement methodology.
m/z	–	The ratio of mass (m) to charge (z) of ions measured by GC/MS.

Post Digestion Spike	–	The addition of a known amount of standard after digestion. (Also identified as analytical spike or spike for furnace analysis).
Primary Analysis	–	One of two types of pesticide/PCB analysis by GC/EC techniques, the other being confirmation analysis. The primary analysis is used to establish the tentative identification of any pesticides/PCBs detected. The identification is confirmed in the confirmation analysis. If the two analyses are done simultaneously, either may be considered the primary analysis. Either may be used for quantitation if contract criteria are met.
QA	–	Quality Assurance – total program for assuring the reliability of data
QC	–	Quality Control – routine application of procedures for controlling the monitoring process.
RL	–	Reporting Limit – value specified by the client based on sensitivity requirements from project-specific action levels.
RPD	–	Relative Percent Difference (between matrix spike and matrix spike duplicate, duplicate laboratory control samples, or blank spikes)
Serial Dilution	–	A sample run at a specific dilution to determine whether any significant chemical or physical interferences exist due to sample matrix effects (ICP only).
SDG	–	Sample Delivery Group – defined by one of the following, whichever occurs first: • Case of field samples • Each 20 field samples within a case • Each 14-day calendar period during which field samples in a case are received, beginning with receipt of the first sample in the SDG
Level B Validation	–	Data validation is performed using sample results and QA/QC summaries (i.e., method blanks, LCS, MS/MSDs, surrogates, and serial dilutions). This level of data validation was previously identified as "Standard."
Level C Data Validation	–	Data validation is performed using sample results and QA/QC summaries (including instrument performance, calibration, and internal standard data). This level of data validation was previously identified as "Cursory."
Level D Data Validation	–	Data validation is performed using sample results, QA/QC summaries (including instrument performance, calibration, and internal standard data) and raw data associated to the sample results and QA/QC summaries. This level of data validation was previously identified as "Full."

Attachment II-A-3
Sample Cover Letter

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SAMPLE COVER LETTER

(Date)

(CTO Manager or designee) (company address) Dear ( ): Enclosed is Revision __ of the data validation reports for CTO (number) as follows: Semi-volatiles SDG S0221 SDG S0350 Pesticides/PCBs SDG S0201 Metals SDG S0221 SDG S0201 The specific sample identifications are listed in the Sample Identification Table(s). The data packages were reviewed according to the data validation procedures referenced in the introduction to each report.

Sincerely,

(Signature)

Data Validation Project Manager

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Attachment II-A-4
Data Qualifier Reference Table

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Table II-A-4-1: Data Qualifier Reference Table

Qualifier	Organics	Inorganics
U	The analyte was analyzed for, but was not detected above the method detection limit.	The analyte was analyzed for, but was not detected above the method detection limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."	Not applicable.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	Not applicable.
UJ	The analyte was not detected above the method detection limit. However, the associated value is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.	The analyte was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.	The data are unusable. The sample results are rejected due to serious deficiencies in meeting the Quality Control (QC) criteria. The analyte may or may not be present in the sample.

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Attachment II-A-5
Qualification Code Reference Table

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Table II-A-5-1: Qualification Code Reference Table

Qualifier	Organics	Inorganics
H	Holding times were exceeded.	Holding times were exceeded.
S	Surrogate recovery was outside QC limits.	The sequence or number of standards used for the calibration was incorrect.
C	Calibration %RSD, r, r^2 or %D were noncompliant	Correlation coefficient is <0.995.
R	Calibration RRF was <0.05.	%R for calibration is not within control limits
B	Presumed contamination from preparation (method blank)	Presumed contamination from preparation (method) blank or calibration blank
L	Laboratory Control Sample/Laboratory Control Sample Duplicate %R or RPD was not within control limits	Laboratory Control Sample/Laboratory Control Sample Duplicate %R or RPD was not within control limits
Q	MS/MSD recovery was poor	MS/MSD recovery was poor.
E	MS/MSD or Duplicate RPD was high.	MS/MSD or Duplicate RPD or difference was high.
I	Internal standard performance was unsatisfactory	ICP ICS results were unsatisfactory.
A	Not applicable.	ICP Serial Dilution %D were not within control limits
M	Instrument Performance Check (BFB or DFTPP) was noncompliant	Not applicable.
T	Presumed contamination from trip blank.	Not applicable.
F	Presumed contamination from FB or ER.	Presumed contamination from FB or ER.
D	The analysis with this flag should not be used because another more technically sound analysis is available.	The analysis with this flag should not be used because another more technically sound analysis is available.
P	Instrument performance for pesticides was poor	Post Digestion Spike recovery was not within control limits
V	Unusual problems found with the data that have been described in the validation report where a description of the problem can be found.	Unusual problems found with the data that have been described in where a description of the problem can be found.

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Attachment II-A-6
Sample Identification Table

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Table II-A-6-1: Sample Identification Table

EPA Identification	Sample Identification	Lab Identification Number	COC Sample Number	Matrix
FB001	FB-BS04-E01-D10.0	2720-1	DA001	water
FB002	FB-BS04-B01-D10.0	2720-2	DA002	water
FB003	FB-BS04-B02-D10.0	2720-3	DA003	water
FB004	FB-SS01-S01-D0.5	2720-4	DA004	soil
FB005	FB-BS01-S01-D10.0	2720-5	DA005	soil
FB006	FB-SS02-S01-D0.5	2720-6	DA006	soil
FB007	FB-BS02-S01-D10.0	2720-7	DA007	soil
FB008	FB-BS02-D01-D10.0	2720-8	DA008	soil
FB009	FB-SS03-S01-D0.5	2720-9	DA009	soil
FB010	FB-BS03-S01-D10.0	2720-10	DA010	soil

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Attachment II-A-7
Example Annotated Laboratory Report
Volatile Organics Analysis Data Sheet

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EXAMPLE ANNOTATED LABORATORY REPORT
VOLATILE ORGANICS ANALYSIS DATA SHEET

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.
CA145

Lab Name: COLUMBIA ANALYTICAL SERVI Contract: EARTH TECH

Lab Code: COLUMB Case No.: SAS No.: SDG No.: K9804746

Matrix: (soil/water) SOIL Lab Sample ID: K9804746-013

Sample wt/vol: 5.1 (g/mL) G Lab File ID: 0727F009

Level: (low/med) LOW Date Received: 07/17/98

% Moisture: not dec. 11 Date Analyzed: 07/27/98

GC Column: RTX-624 ID: 0.32 (mm) Dilution Factor: 1.0

Soil Extract Volume:_____(uL) Soil Aliquot Volume: _____(uL)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/KG	Q
74-87-3	Chloromethane	11	U u
74-83-9	Bromomethane	11	U
75-01-4	Vinyl Chloride	11	U
75-00-3	Chloroethane	11	U
75-09-2	Methylene Chloride	0.8	JB nu (B)
67-64-1	Acetone	2	JB L
75-15-0	Carbon Disulfide	11	U u
75-35-4	1,1-Dichloroethene	11	U
75-34-3	1,1-Dichloroethane	11	U
540-59-0	1,2-Dichloroethene (total)	11	U
67-66-3	Chloroform	11	U
107-06-2	1,2-Dichloroethane	11	U
78-93-3	2-Butanone	11	U
71-55-6	1,1,1-Trichloroethane	11	U
56-23-5	Carbon Tetrachloride	11	U
75-27-4	Bromodichloromethane	11	U
78-87-5	1,2-Dichloropropane	11	U
10061-01-5	cis-1,3-Dichloropropene	11	U
79-01-6	Trichloroethene	11	U
124-48-1	Dibromochloromethane	11	U
79-00-5	1,1,2-Trichloroethane	11	U
71-43-2	Benzene	11	U
10061-02-6	trans-1,3-Dichloropropene	11	U
75-25-2	Bromoform	11	U
108-10-1	4-Methyl-2-Pentonone	11	U
591-78-6	2-Hexanone	11	U
127-18-4	Tetrachloroethene	0.2	J J
79-34-5	1,1,2,2-Tetrachloroethane	11	U u
108-88-3	Toluene	0.2	JB nu (B)
108-90-7	Chlorobenzene	11	U u
100-41-4	Ethylbenzene	11	U u
100-42-5	Styrene	0.4	JB nu (B)
1330-20-7	Xylene (Total)	0.2	JB nu (B)

FORM I VOA

01513

"U.S. NAVY PACDIV IRP VALIDATED"

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Level C and Level D Data Validation for GC/MS Volatile Organics by SW-846 8260

1. Purpose

This data validation procedure sets forth the standard operating procedure for performance of Level C and Level D data validation of volatile organic data obtained under the United States (U.S.) Navy Environmental Restoration (ER) Program for Naval Facilities Engineering Command (NAVFAC), Pacific and is consistent with protocol in the *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories* (DoD QSM) (DoD 2013). Level B validation is addressed separately in Procedure II-A, *Data Validation*.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Responsibilities

The CTO Manager, the QA Manager or Technical Director, and the CTO QA Coordinator are responsible for ensuring that this procedure is implemented by data validation personnel.

Data validation personnel are responsible for implementing this procedure for validation of all gas chromatography/mass spectrometry (GC/MS) volatile data.

4. Procedure

This procedure addresses the validation of volatile organic data obtained using U.S. Environmental Protection Agency Method Solid Waste (SW)-846 8260 (EPA 2007). The quality control (QC) criteria identified in this procedure are those specified in the analytical method and the DoD QSM (DoD 2013). Where project specific criteria are identified in the CTO work plan, they will supersede the QC criteria identified in this procedure.

- Form I: Sample Results Summary Form
- Form II: Surrogate Recovery Summary Form
- Form III: Matrix Spike/Matrix Spike Duplicate or Blank Spike/Blank Spike Duplicate Recovery Summary Form

- Form IV: Method Blank Summary Form
- Form V: Instrument Performance Check Summary Form
- Form VI: Initial Calibration Summary Form
- Form VII: Continuing Calibration Summary Form
- Form VIII: Internal Standard Summary Form

Level C data validation consists of review of summary forms only, whereas Level D data validation requires review of both summary forms and all associated raw data. Data review guidelines and how they apply to the different validation levels are indicated in the following text.

4.1 SAMPLE MANAGEMENT

QA/QC criteria included under sample management are sample preservation, handling, and transport, chain of custody (COC), and holding times.

4.1.1 Sample Preservation, Handling, and Transport

Level C and Level D:

Evaluate sample collection, handling, transport, and laboratory receipt from COC and laboratory receipt checklists to ensure that the samples have been properly preserved and handled.

1. Water samples must be preserved with hydrochloric acid at or below a pH of 2 and refrigerated at above freezing to 6 degrees Celsius (°C).
2. Soil samples collected in volatile organic analysis (VOA) vials or coring devices must be refrigerated at or above freezing to 6°C. If the samples are to be analyzed after the 48-hour holding time, the laboratory must preserve the samples with sodium bisulfate or methanol or water or freeze upon receipt in accordance with SW-846 Method 5035.
3. If the analyzed aqueous VOA vial contains air bubbles or headspace, is cracked, or has a cracked cap, positive values shall be flagged as estimated "J" and nondetects as estimated "UJ." The sample data may be qualified as unusable, "R," if the container damage is extensive or improper sealing is identified.
4. VOA vials are to be shipped in coolers that are maintained at above freezing to 6°C. If the temperature exceeds 6°C, but is less than or equal to 10°C, note this in the data validation report. If the temperature of receipt is greater than or equal to 11°C, positive values shall be flagged as estimated "J" and nondetects as estimated "UJ." If the temperature of receipt is greater than or equal to 15°C, positive values shall be flagged as estimated "J" and nondetects as unusable "R." If the temperature is below 0°C, special note should be made that the samples were frozen and no qualification shall be required. In the event that both a cooler temperature and a temperature blank were measured, the temperature blank shall be evaluated for temperature compliance as it best assimilates the condition of the samples; however, both temperatures shall be noted in the data validation report.
5. If the temperature of the cooler upon receipt at the laboratory was not recorded, document that the laboratory is noncompliant.

6. If the receiving laboratory transferred the samples to another laboratory for analysis, apply the same temperature criteria to both the transfer COC and the original COC.

4.1.2 Chain of Custody

Level C and Level D:

Examine the COC form for legibility and check that all volatile analyses requested on the COC have been performed by the laboratory. Ensure that the COC Sample Number on the laboratory sample results form (Form I [or equivalent]) matches the Sample Identification on the COC. Read the laboratory case narrative for additional information.

1. Any samples received for analysis that were not analyzed shall be noted in the data validation report, along with the reason(s) for failure to analyze the samples, if the reason(s) can be determined. Conversely, samples that were analyzed for volatiles but were not requested should also be noted.
2. Any discrepancies in sample naming between the COC and sample results form shall be noted in the data validation report with the correct sample name being identified if the correct sample name can be determined.
3. If the receiving laboratory transferred the samples to another laboratory for analysis, both the original COCs and transfer COCs shall be present. Document in the data validation report if the transfer COCs are not present.
4. Internal COC is required for all samples, extracts, and digestates from receipt to disposal. Verify the internal COC forms for completeness. Document in the data validation report if the internal COC forms are not present.
5. Each individual cooler shall have an individual COC that lists only samples contained within that cooler. Document in the data validation report if multiple coolers appear on one COC.

4.1.3 Holding Times

Level C and Level D:

Holding times for volatile organics are measured from the time of collection (as shown on the COC) to the time of sample analysis (as shown on the sample results form and instrument performance check summary form [Forms I and V (or equivalent)]). Water samples must be preserved with hydrochloric acid and refrigerated at above freezing to 6°C. Preserved water samples shall be analyzed within 14 days from the collection date. If there is no indication of chemical preservation, assume samples are unpreserved. For unpreserved water samples, the holding time is 7 days from date collected for aromatic volatiles and 14 days from date collected for non-aromatic volatiles. Soil samples collected in VOA vials or coring devices that are unpreserved must be refrigerated at above freezing to 6°C and analyzed within 48 hours from the collection date. Soil samples that are preserved with sodium bisulfate or methanol, or frozen upon laboratory receipt shall be analyzed within 14 days from the collection date.

1. If the holding time is exceeded, flag all associated positive results as estimated "J" and all associated limits of detection (LODs) (nondetects) as estimated "UJ," and document that holding times were exceeded.

2. If holding times are grossly exceeded by greater than a factor of 2.0 (e.g., a preserved water sample has a holding time of more than 28 days), detects will be qualified as estimated "J" and nondetects as unusable "R."

4.2 GC/MS INSTRUMENT PERFORMANCE CHECK

Level C and Level D:

GC/MS instrument performance checks or tune checks are performed to ensure mass resolution, identification, and to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard reference materials; therefore, these criteria should be met in all circumstances.

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, bromofluorobenzene (BFB) for volatile analysis, must meet the ion abundance criteria given below.

Table II-B-1: Ion Abundance Criteria – BFB

m/z	Ion Abundance Criteria
50	15.0–40.0% of m/z 95
75	30.0–60.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0–9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	Greater than 50.0% of m/z 95
175	5.0–9.0% of m/z 174
176	Greater than 95.0% but less than 101.0% of m/z 174
177	5.0–9.0% of m/z 176

% percent
m/z mass-to-charge ratio

Check that all sample runs are associated with an injection. Make certain that a BFB performance check is present for each 12-hour period samples are analyzed (Form V [or equivalent]). Verify that all samples were analyzed within 12 hours of BFB injection.

If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. The most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and type of instrumentation; therefore, the critical ion abundance criteria for BFB are the mass-to-charge ratio (m/z) 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundance of m/z 50 and 75 are of lesser importance. Use professional judgment when samples are analyzed beyond the 12-hour time limit.

Decisions to use analytical data associated with BFB instrument performance checks not meeting requirements should be noted in the data validation report.

Level D:

Verify by recalculating from the quantitation reports, mass spectra, and chromatograms that the mass assignment is correct and that the mass listing is normalized to the specified m/z. If transcription errors are discovered on the Form V (or equivalent), request a resubmittal from the laboratory. Validate the data using the criteria outlined above.

4.3 CALIBRATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the volatile target compound list.

4.3.1 Initial Calibration

Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing an acceptable calibration curve.

Level C and Level D:

1. Evaluate the average relative response factors (RRFs) for all target compounds by checking Form VI (or equivalent).
2. If any of the volatile target compounds listed in Table II-B-2 below has an average RRF of less than 0.01 except for 1,4-dioxane (≤0.005) or any of the other volatile target compounds has an average RRF of less than 0.05, flag positive results for that compound as estimated "J" and nondetects as unusable "R" in associated samples.

Table II-B-2: Volatile Compounds Exhibiting Poor Response

Acetone	1,2-Dibromo-3-chloropane
2-Butanone	Isopropylbenzene
Carbon disulfide	Methyl acetate
Chloroethane	Methylene chloride
Chloromethane	Methylcyclohexane
Cyclohexane	Methyl tert-butyl ether
1,2-Dibromoethane	trans-1,2-Dichloroethene
Dichlorodifluoromethane	4-Methyl-2-pentanone
1,2-Dichloropropane	2-Hexanone
cis-1,2-Dichloroethene	Trichlorofluoromethane
1,4-Dioxane	1,1,2-Trichloro-trifluoromethane

3. Check Form VI (or equivalent) and evaluate the percent relative standard deviation (%RSD) for all target compounds. If any volatile target compound has a %RSD of greater than 15 percent, flag detects for the affected compounds as "J" and nondetects as "UJ" in the associated samples that correspond to that initial calibration.

Level D:

1. Verify the files reported on Form VI (or equivalent) against the quantitation reports, mass spectra, and chromatograms. If the files do not match, the RRFs reported are likely to be

from another initial calibration and will have to be changed. Request a resubmittal from the laboratory.

2. Recalculate the average RRFs and %RSDs reported on Form VI (or equivalent) for one compound per internal standard (preferably compounds which were identified in the samples) on the low-point calibration standard and one additional calibration standard. If errors are discovered, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.3.2 Initial Calibration Verification

The initial calibration curve must be verified with a standard that has been purchased or prepared from an independent source each time initial calibration is performed. A standard from the same manufacturer but independently prepared from different source materials may also be used as an independent source. This initial calibration verification (ICV) must contain all of the method target compounds.

Level C and Level D:

1. Verify the ICV was analyzed following the initial calibration and contained all method target compounds.

2. If any target analyte has a percent difference (%D) greater than 20 percent, flag detects for the affected compounds as estimated "J" and nondetects as estimated "UJ" in all samples associated with the initial calibration.

Level D:

1. Verify from the raw data that there were no calculation or transcription errors by recalculating a percentage of the ICV calculations.

4.3.3 Continuing Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

Level C and Level D:

1. Continuing calibration standards containing both target compounds and system monitoring compounds must be analyzed every 12 hours during operation. Evaluate the continuing RRFs on Form VII (or equivalent).
2. Ensure that the average RRFs reported on Form VII (or equivalent) correspond to the average RRFs reported on Form VI (or equivalent) for the corresponding initial calibration.
3. If any of the volatile target compounds listed in Table II-B-2 has an average RRF of less than 0.01 except for 1,4-dioxane (≤0.005) or any of the other volatile target compounds has an average RRF of less than 0.05, flag positive results for that compound as estimated "J" and nondetects as unusable "R" in associated samples.

4. If any volatile target compound has a %D between the initial calibration average RRF and continuing calibration RRFs outside 20 percent, flag all detects as "J" and all nondetects as "UJ" in all associated samples that correspond to that continuing calibration.

5. An ending continuing calibration is required by DoD QSM Appendix B (an ending continuing calibration is not required by the method) and professional judgment should be used in qualifying associated data when the %D is outside 50 percent.

Level D:

1. Verify the file reported on Form VII (or equivalent) against the raw data for the continuing calibration. If the file does not match, the RRFs reported are likely to be from another continuing calibration and will have to be changed. Request a resubmittal from the laboratory.
2. Recalculate the reported RRFs and %Ds reported on Form VII (or equivalent) for one compound per internal standard. If errors are discovered, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.4 BLANKS

Method blank analytical results are assessed to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all associated data must be carefully evaluated to determine whether there is any bias associated with the data, or if the problem is an isolated occurrence not affecting other data. Results may not be corrected by subtracting any blank values.

Level C and Level D:

1. The reviewer should identify samples associated with each method blank using Form IV (or equivalent). Verify that method blank analysis has been reported per matrix and concentration level for each 12-hour time period on each GC/MS system used to analyze VOA samples. Each sample must have an associated method blank. Medium level samples (samples that are known to have high concentrations of compounds) should have an associated methanol extraction blank. Qualify positive results in samples with no method blank as unusable "R." Nondetects do not require qualification.
2. Compare the results of each method blank with the associated sample results. The reviewer should note that the blank analyses may not involve the same weights, volumes, percent moistures, or dilution factors as the associated samples. These factors must be taken into consideration when applying the criteria discussed below, such that a comparison of the total amount of contamination is actually made.
3. If a compound is found in the blank, but not in the associated sample, no action is taken.
4. Any compound, other than those listed in Table II-B-3, detected in both the sample and the associated blank shall be qualified when the sample concentration is less than the limit of quantitation (LOQ) and the blank concentration is less than, greater than, or equal to the LOQ. Compounds listed in Table II-B-3 shall be qualified when the sample concentration is less than 2× the LOQ and the blank concentration is less than, greater than, or equal to 2× LOQ. Care should be taken to factor in the percent moisture when comparing detects in the

sample and the method blank. The applicable review qualifier(s) are summarized in Table II-B-4.

Table II-B-3: Common Laboratory Contaminants

1. Methylene chloride
2. Acetone
3. 2-Butanone

Table II-B-4: Blank Qualifications

Sample Result	Sample Value	Reviewer Qualifier(s)
Less than LOQ* and blank result is <, > or = LOQ*	Leave as reported	U
≥LOQ*, blank result is <LOQ*	Leave as reported	None
≥LOQ*, blank result is >LOQ* and sample result < blank result	Leave as reported	Use professional judgment
≥LOQ*, blank result is >LOQ* and sample result ≥ blank result	Leave as reported	Use professional judgment
≥LOQ* and blank result is = LOQ*	Leave as reported	Use professional judgment

* 2x LOQ for common laboratory contaminants

5. In the case wherein both the sample concentration and the blank concentration are greater than or equal to the LOQ, previously approved criteria as identified in the planning documents may be applied to qualify associated sample results. Otherwise qualify sample results as non-detect "U" when the sample concentration is less than or equal to 10 times the blank concentration (10× rule) for the compounds listed in Table II-B-3 and tentatively identified compounds (TICs). For all other compounds, qualify sample results as non-detect "U" when the sample concentration is less than or equal to 5 times the blank concentration (5× rule).
6. If gross contamination exists in the blanks (i.e., saturated peaks by GC/MS), all compounds affected shall be flagged as unusable "R" due to interference in all samples affected and this shall be noted in the data validation comments.
7. If target compounds other than common laboratory contaminants are found at low levels in the blank(s), it may be indicative of a problem at the laboratory and shall be noted in the data validation report.
8. Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. It may be impossible to verify this source of contamination; however, if the reviewer determines that the contamination is from a source other than the sample, the data should be qualified The sample value shall be reported as a nondetect and the reason shall be documented in the data validation report. Qualification of the data will be performed as given in Table II-B-4.

Level D:

1. Verify all target compound and TIC detects found in the method blanks against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid or the tentative identity is in error, request for a corrected Form I for the method blank from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

Blank spike/laboratory control sample (LCS) recoveries must be within the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established for a given sample matrix. Use in-house limits if compounds are not listed in Appendix C or project limits are not specified.

Level C and Level D:

1. If the blank spike/LCS results are 0 percent, only the spiked compounds that showed low recovery in all associated samples shall be flagged as unusable "R" for nondetects and estimated "J" for detects.
2. If blank spike/LCS results are below the control limits (but above 0 percent), spiked compounds which showed low recovery in all associated samples shall be flagged as estimated "UJ" or "J."
3. If blank spike/LCS results are above the control limits, detects for only the spiked compounds which showed high recovery in all associated samples shall be flagged as estimated "J."
4. If the laboratory analyzes a blank spike duplicate/LCS duplicate (LCSD), evaluate and qualify the LCSD results using the criteria noted above.
5. If the relative percent differences (RPDs) between LCS and LCSD results are above the control limits (use the matrix spike [MS]/matrix spike duplicate [MSD] RPD control limits identified in DoD QSM Appendix B. if none are available use laboratory in-house limits), spiked compounds which showed high RPD in all associated samples shall be flagged as estimated "UJ" or "J."

Level D:

To check that the spike percent recovery was calculated and reported correctly using the following equation, recalculate one or more spike recoveries per matrix (and any spike that would result in the qualification of a sample).

$$\%\text{Recovery} = \frac{Q_D}{Q_A} \times 100$$

Where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.6 System Monitoring Compounds (Surrogate Spikes)

Laboratory performance on individual samples is established by means of surrogate spiking activities. All samples are spiked with surrogate compounds prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interference and high concentrations of compounds. Because the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. These procedures shall be followed:

Level C and Level D:

1. Sample and blank surrogate recoveries for volatiles must be within the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established. Use in-house limits if surrogates are not listed in Appendix C or project limits are not specified. Verify that no samples or blanks have surrogates outside the criteria from Form II (or equivalent).
    a. If one surrogate for the VOA fraction is out of specification, then a re-analysis must be reported even though surrogate results are outside the criteria. (Note: When unacceptable surrogate recoveries are followed by successful re-analyses, the laboratory is required to report only the successful run. The laboratory does not have to re-analyze a sample if a MS/MSD was performed on the sample with out-of-control surrogate results showing the same matrix effects.) Medium level soils must be re-extracted and re-analyzed if the surrogate recoveries are outside the criteria.
    b. The laboratory has failed to perform satisfactorily if surrogate recoveries are out of specification with no evidence of re-purging. The non-surrogate recoveries shall be documented in the data validation report.
2. If surrogate spike recoveries are out of specification, samples will be qualified as follows:
    a. If any surrogate is below the lower acceptance limit but has a recovery greater than or equal to 10 percent, qualify positive results as estimated "J" and nondetects as estimated "UJ."
    b. If any surrogate is above the upper acceptance limit, qualify detects in the sample as estimated "J." Compounds with nondetects should not be qualified.

3. If any surrogate in a fraction shows less than 10 percent recovery, flag detects for that fraction as estimated "J," and nondetects for the fraction as unusable "R."
4. In the special case of blank analysis with surrogates out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if the samples in the batch show acceptable surrogate recoveries, the reviewer may determine the blank problem to be an isolated occurrence for which no qualification of the data is required.
5. Surrogates may be reported as "diluted out" (D), if dilution is such that the surrogate can no longer be detected. If this is the case, note in the data validation report that surrogate evaluation could not be performed due to a high dilution factor. A full evaluation of the sample chromatogram and quantitation report may be necessary to determine that surrogates are truly "diluted out."

Level D:

To verify that the surrogate percent recovery was calculated and reported correctly using the following equation, recalculate all surrogate recoveries per matrix (and any surrogate that would result in the qualification of a sample).

$$\%\text{Recovery} = \frac{Q_D}{Q_A} \times 100$$

Where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

If transcription errors are discovered on Form II (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD data are used to determine the effect of the matrix on a method's recovery efficiency and precision for a specific sample matrix.

No action is taken on MS/MSD data alone to qualify an entire data package. Using informed professional judgment; however, the data reviewer may use the MS/MSD results in conjunction with other QC criteria (i.e., surrogates and LCS) and determine the need for some qualification of the data.

In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. It may be determined through the MS/MSD results, however, that a laboratory is having a systematic problem in the analysis of one or more compounds, which affects all associated samples.

If a field blank was used for the MS/MSD, the information must be included in the data validation summary. Sample matrix effects have not been observed with field blanks therefore the recoveries and precision do not reflect the analytical impact of the site matrix.

Level C and Level D:

The laboratory must spike and analyze an MS/MSD from the specific project site as required for each matrix type and analytical batch.

1. MS/MSD data should be reported on a MS/MSD summary form similar to Form III (or equivalent).
2. Compare the percent recovery (%R) and RPD for each spiked compound with the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established. Use in-house limits if spiked compounds are not listed in Appendix C or project limits are not specified.
3. If MS/MSD results are 0 percent, only the spiked compounds that showed low recovery in the parent sample shall be flagged as unusable "R" for nondetects and estimated "J" for detects.
4. If MS/MSD results are below the control limits (but above 0 percent), spiked compounds which showed low recovery in the parent sample shall be flagged as estimated "UJ" or "J."
5. If MS/MSD results are above the control limits, detects for only the spiked compounds which showed high recovery in the parent sample shall be flagged as estimated "J."
6. If the RPDs between MS and MSD results are greater than 20 percent, detects for only the spiked compounds which showed high RPD in the parent sample shall be flagged as estimated "J."
7. Failure of MS/MSD due to the presence of a target compound in the parent sample at greater than two times the spike concentration or diluted by more than a factor of 2 should not result in any qualifications. Note the incident in the data validation report.

Level D:

Check the raw data and recalculate one or more %Rs and RPDs, especially %Rs and RPDs that resulted in the qualification of data, using the following equations to verify that results on Form III (or equivalent) are correct.

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

$$RPD = \frac{ABS|SSR - SDR|}{(SSR + SDR)/2} \times 100$$

Where:

SA	=	spike added
SR	=	sample result
SSR	=	spiked sample result
SDR	=	spiked duplicate result
ABS	=	absolute value

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.8 FIELD QC SAMPLES

Field QC samples discussed in this section of this procedure are trip blanks, equipment blanks, field blanks, field duplicates, and field triplicates.

4.8.1 Trip Blanks

Volatile organic parameters detected in trip blanks indicate the possibility of contamination of site samples or cross-contamination between site samples due to sample handling and transport while in the cooler.

One trip blank shall accompany each cooler containing samples to be analyzed for volatile organics. Each trip blank shall be analyzed for all volatile organic parameters for which the associated samples are analyzed. If a cooler contains multiple trip blanks, all samples contained in the cooler shall be associated with the results from all trip blanks contained in the cooler.

Level C and Level D:

1. Check that all coolers containing samples to be analyzed for volatile organics contained a trip blank that was also analyzed for volatile organics. If a cooler requiring a trip blank did not have an associated trip blank, no qualification of the samples transported in the cooler is necessary, but the incident shall be discussed in the data validation report.
2. If volatile organic compounds are detected in the trip blanks, the procedure for the qualification of associated sample results using validated and/or qualified trip blank results is identical to the criteria outlined in Section 4.4 of this procedure.

Level D:

1. Verify all target compound and TIC detects found in the trip blanks against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and verify the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid, or the tentative identity is in error, request for a corrected Form I (or equivalent) for the trip blank from the laboratory.

3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.8.2 Equipment Blanks and Field Blanks

1. Compounds detected in equipment blanks indicate the possibility of cross-contamination between samples due to improper equipment decontamination.
2. A field blank sample may be collected from each source of water used during each sampling event. The field blank may be analyzed to assess whether the chemical nature of the water used in decontamination may have affected the analytical results of site samples.
3. If volatile organic compounds are detected in the equipment blanks and/or field blanks, the procedure for the qualification of associated sample results is identical to the criteria outlined in Section 4.4 of this procedure.

Level C and Level D:

1. Determine which field QC samples apply to samples in the sample delivery group (SDG).
2. Ensure that units are correct when applying field QC blank qualifications. If samples are soil matrix, results must first be converted to micrograms per liter (µg/L) from milligrams per kilogram to make correct comparisons.
3. Because of the way in which the field blanks and equipment blanks are sampled, equipment blanks are not qualified because of field blank contamination. The affected samples are qualified, however, by either the field blank or equipment blank results, whichever has the higher contaminant concentration.
4. Equipment blanks and field blanks are only qualified with method and trip blank results in order to account for laboratory contamination.

Level D:

1. Verify all target compound and TIC detects found in the equipment blanks and field blanks against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid, or if the tentative identity is in error, request for a corrected Form I (or equivalent) for the equipment blank or field blank from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.8.3 Field Duplicates and Field Triplicates

Field duplicates consist of either collocated or subsampled samples. Field duplicates for ground water and surface water samples are generally considered to be collocates. Soil duplicate samples may be homogenized and subsampled in the field (or at the laboratory) to form an original and duplicate sample, or may be an additional volume of sample collected in a separate sample container

to form a collocate sample. Field duplicate results are an indication of both field and laboratory precision; the results may be used to evaluate the consistency of sampling practices.

Field triplicates are collected from different, randomly selected locations to verify that an incremental sample truly represents a decision unit. Field triplicate results are more useful than field duplicates to statistically evaluate sampling precision.

Level C and Level D:

1. Check to ensure that field duplicates and/or field triplicates were collected and analyzed as specified in the project planning documents. If the sampling frequency is less than the frequency stated in the planning documents, no qualification of the associated sample results is necessary but the incident shall be discussed in the data validation report.
2. For field duplicate results, if the RPDs are greater than 50 percent for water or 100 percent for soil or as stated in the planning document if more conservative, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.
3. For field triplicate results, if the RSDs are greater than the QC limits stated in the planning document, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.

Level D:

1. Verify all target compound and TIC detects found in the field duplicates and/or field triplicates against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid, or if the tentative identity is in error, request for a corrected Form I for the field duplicates from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.9 INTERNAL STANDARDS PERFORMANCE

Internal standards performance criteria ensure that GC/MS sensitivity and response are stable during every analytical run.

Level C and Level D:

1. If an internal standards area count for a sample is outside –50 percent or +100 percent of the area for the initial calibration midpoint standard:
   a. Positive results for compounds quantitated using an internal standards area count greater than 100 percent should be qualified as estimated "J." Nondetected compounds should not be qualified.

b. Compounds quantitated using an internal standards area count less than 50 percent should be qualified as estimated "J" for detects and estimated "UJ" for nondetects.

c. If extremely low area counts are reported (less than 20 percent of the area for associated standards), detected compounds should be qualified as estimated "J" and nondetected target compounds should then be qualified as unusable "R."

2. If an internal standards retention time (RT) varies by more than 10 seconds from the RT of the initial calibration midpoint standard, the nondetected target compounds should be qualified as unusable "R" at Level C validation. A Level D validation examination of the raw data should be recommended to the CTO Manager. The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Positive results should be qualified as "NJ" if the mass spectral criteria are met.

Level D:

1. Verify the internal standard areas reported on Form VIII (or equivalent) from the raw data for at least one sample per SDG, and verify internal standard areas for samples that were qualified due to out-of-control internal standard areas. If errors are discovered between the raw data and the Form VIII (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.10 TARGET COMPOUND IDENTIFICATION

The objective of the criteria for GC/MS qualitative analysis is to minimize the number of erroneous identifications of target compounds. An erroneous identification can either be false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

The identification criteria can be applied more easily in detecting false positives than false negatives. More information is available for false positives due to the requirement for submittal of data supporting positive identifications. However, negatives, or nondetected compounds, represent an absence of data and are therefore more difficult to assess. One example of detecting false negatives is the not reporting of a target compound that is reported as a TIC.

Level C:

Target compound identification is not evaluated for Level C validation since it requires the interpretation of mass spectral raw data.

Level D:

The following criteria should be followed when evaluating raw data.

1. The relative retention times (RRTs) must be within ± 0.06 RRT units of the standard RRT.
2. Mass spectra of the sample compound and a current laboratory-generated standard (i.e., the mass spectrum from the associated calibration standard) must match according to the following criteria:

a. All ions present in the standard mass spectrum at a relative intensity greater than 10 percent must be present in the sample spectrum.

b. The relative intensities of these ions must agree within ± 20 percent between the standard and sample spectra. (Example: For an ion with an abundance of 50 percent in the standard spectrum, the corresponding sample ion abundance must be between 30 percent and 70 percent.)

c. Ions present at greater than 10 percent in the sample mass spectrum, but not present in the standard spectrum, must be considered and accounted for.

d. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory and CTO Manager. If it is determined that incorrect identifications were made, all such data should be qualified as not detected "U" or unusable "R."

e. Professional judgment must be used to qualify the data if it is determined that cross-contamination has occurred. Any changes made to the reported compounds or concerns regarding target compound identifications should be clearly indicated in the data validation report.

4.11 Compound Quantitation and Reporting Limits

The objective is to ensure that the reported quantitation results and reporting limits (i.e., LOQ, LOD, detection limit [DL]) are accurate. All soil sample results are reported on a dry weight basis.

Level C and Level D:

1. Verify that the reporting limits for nondetects are equal to the LODs. Verify that an annual DL study was performed or quarterly LOD/LOQ verification checks were performed in accordance with the DoD QSM. The LOD verification check must be evaluated to determine whether the laboratory can reliably detect and identify all target analytes at a spike concentration of approximately 2× but not more than four times the current reported DL. Qualify nondetects as unusable "R."

2. Check that reported nondetects and positive values have been adjusted to reflect sample dilutions and for soil samples, sample moisture. When a sample is analyzed at more than one dilution, the lowest LODs are used unless a QC criterion has been exceeded. In this case, the higher LODs from the diluted analysis are used. The least technically sound data will be flagged "R" with a qualification code "D."

3. Verify that reported limits for soils and sediments were calculated based on dry weight. If the LOQs/LODs were reported based on wet weight, the percent moisture must be factored in and the LOQs/LODs must be adjusted accordingly.

4. Verify that no results exceed the highest calibration standard without being diluted. If a result has exceeded the highest calibration standard, verify that a dilution was performed. If not, qualify the detected compound that required dilution as "J" and document the event in the data validation report.

Level D:

The compound quantitation must be evaluated for all detects by evaluating the raw data. Compound concentrations must be calculated based on the internal standards associated with that compound, as listed in the following equation. Quantitation must be based on the quantitation ion (m/z) specified in the analytical method for both the internal standards and target compounds. The compound quantitation must be based on the RRF from the appropriate ICAL standard.

Low Water

$$\mu g/L = \frac{A_x \times I_s \times D_f}{A_{is} \times ARRF \times V_o}$$

Where:

A_x	=	area of characteristic ion (extracted ion current profile) for compound being measured
I_s	=	amount of internal standard added (nanogram)
D_f	=	dilution factor
A_{is}	=	area of characteristic ion for the internal standard
ARRF	=	average relative response factor for compound being measured
V_o	=	volume of water purged (milliliter [mL])

Low Soil/Sediment

$$\text{Concentration } \mu g/kg \text{ (Dry weight basis)} = \frac{A_x \times I_s}{A_{is} \times ARRF \times W_s \times D}$$

Where:

A_x, I_s, A_{is} are as given for water.

ARRF	=	Relative response factor from the heated purge of the initial calibration standard
W_s	=	Weight of sample added to the purge tube, in grams (g)
D	=	$\frac{100 - \%\text{ moisture}}{100}$

Medium Soil/Sediment

$$\text{Concentration } \mu g/kg \text{ (Dry weight basis)} = \frac{A_x \times I_s \times V_t \times 1{,}000 \times D_f}{A_{is} \times ARRF \times V_a \times W_s \times D}$$

Where:

A_x, I_s, A_{is}, D are as given for water.

V_t = Total volume of the methanol extract in mL. Note: This volume is typically 10 mL, even though only 1 mL is transferred to the vial

ARRF = Average relative response factor from the ambient temperature purge of the initial calibration standard

V_a = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 microliters [µL]) in µL added to reagent water for purging

W_s = Weight of soil/sediment extracted, in grams (g)

D_f = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by medium level method is defined as:

$$\frac{\mu L \text{ most conc. extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most conc. extract used to make dilution}}$$

The dilution factor is equal to 1.0 in all cases other than those requiring dilution of the sample methanol extract (V_t). The factor of 1,000 in the numerator converts the value of V_t from mL to µL.

If discrepancies are discovered in the quantitation, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.12 TENTATIVELY IDENTIFIED COMPOUNDS

For each sample, the laboratory must conduct a mass spectral search of the spectral library and report the possible identity for up to 30 of the largest volatile fraction peaks that are not system monitoring compounds (surrogates), internal standards, or target compounds, but which have area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I VOA-TIC [or equivalent]).

Level C and Level D:

1. All TIC results should be qualified "NJ," tentatively identified with approximated concentrations.
2. The reviewer should be aware of common laboratory artifacts and their sources such as siloxane compounds, which indicate capillary column degradation, and carbon dioxide which indicates a possible air leak in the system. These may be qualified as unusable "R."
3. If a target compound is identified as a TIC by non-target library search procedures, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion.
4. TIC results that are not above the 10× level in the blank should be qualified as unusable, "R." (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)

5. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).

Level D:

Check each TIC for each sample using the following criteria.

1. Major ions (greater than 10 percent relative intensity) in the reference spectrum should be present in the sample spectrum.
2. The relative intensities of the major ions should agree within ± 20 percent between the sample and the reference spectra.
3. Molecular ions present in the reference spectrum should be present in the sample spectrum.
4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference, or co-elution of additional TIC or target compounds.
5. When the above criteria are not met, but in the technical judgment of the data reviewer or mass spectral interpretation specialist, the identification is correct, the data validator may report the identification.
6. Since TIC library searches often yield several candidate compounds having a close matching score, all reasonable choices must be considered. The reviewer may use judgment to change the reported tentative identity.

5. Records

A Form I or equivalent that has been validated and verified, and has been determined by the data validator to accurately represent the appropriate sample results to be utilized, shall be stamped "NAVFAC PACIFIC VALIDATED." Additionally, sample result forms for which the data has been validated at the Level D validation level shall be stamped or noted "Level D."

Copies of all documents generated by the data validation personnel will be stored for no less than 10 years. The original validated laboratory data shall be archived to the Federal Records Center at project completion.

6. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www2.epa.gov/sites/production/files/documents/qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Environmental Protection Agency, United States (EPA). 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Final Update IV. Office of Solid Waste. On-line updates at: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

Procedure II-A, *Data Validation*.

7. Attachments

None.

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Level C and Level D Data Validation for GC/MS Semivolatile Organics by SW-846 8270 (Full Scan and SIM)

1. Purpose

This data validation procedure sets forth the standard operating procedure for performance of Level C and Level D data validation of semivolatile organic data obtained under the United States (U.S.) Navy Environmental Restoration (ER) Program for Naval Facilities Engineering Command (NAVFAC), Pacific and is consistent with protocol in the *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories* (DoD QSM) (DoD 2013). Level B validation is addressed separately in Procedure II-A, *Data Validation*.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Responsibilities

The CTO Manager, the QA Manager or Technical Director, and the CTO QA Coordinator are responsible for ensuring that this procedure is implemented by data validation personnel.

Data validation personnel are responsible for implementing this procedure for validation of all gas chromatography/mass spectrometry (GC/MS) semivolatile data.

4. Procedure

This procedure addresses the validation of semivolatile organic data obtained using U.S. Environmental Protection Agency (EPA) Method Solid Waste (SW)-846 8270 (EPA 2007). The quality control (QC) criteria identified in this procedure are those specified in the analytical method and the DoD QSM (DoD 2013). Where project specific criteria are identified in the CTO work plan, they will supersede the QC criteria identified in this procedure.

- Form I: Sample Results Summary Form
- Form II: Surrogate Recovery Summary Form
- Form III: Matrix Spike/Matrix Spike Duplicate or Blank Spike/Blank Spike Duplicate Recovery Summary Form

- Form IV: Method Blank Summary Form
- Form V: Instrument Performance Check Summary Form
- Form VI: Initial Calibration Summary Form
- Form VII: Continuing Calibration Summary Form
- Form VIII: Internal Standard Summary Form

Level C data validation consists of review of summary forms only while Level D data validation requires review of both summary forms and all associated raw data. Data review guidelines and how they apply to the different validation levels are indicated in the following text.

4.1 SAMPLE MANAGEMENT

QA/QC criteria included under sample management are sample preservation, handling, and transport; chain of custody (COC); and holding times.

4.1.1 Sample Preservation, Handling, and Transport

Level C and Level D:

Evaluate sample collection, handling, transport, and laboratory receipt from COC and laboratory receipt checklists to ensure that the samples have been properly preserved and handled.

1. Samples are to be shipped in coolers that are maintained at above freezing to 6 degrees Celsius (°C). If the temperature exceeds 6°C but is less than or equal to 10°C, note this in the data validation report. If the temperature of receipt is greater than or equal to 11°C, positive values shall be flagged as estimated "J" and nondetects as estimated "UJ." If the temperature is below 0°C, special note should be made that the samples were frozen and no qualification shall be required. In the event that both a cooler temperature and a temperature blank were measured, the temperature blank shall be evaluated for temperature compliance as it best assimilates the condition of the samples; however, both temperatures shall be noted in the data validation report.
2. If the temperature of the cooler upon receipt at the laboratory was not recorded, document that the laboratory is noncompliant.
3. If the receiving laboratory transferred the samples to another laboratory for analysis, apply the same temperature criteria to both the transfer COC and the original COC.

4.1.2 Chain of Custody

Level C and Level D:

Examine the COC for legibility and check that all semivolatile analyses requested on the COC have been performed by the laboratory. Ensure that the COC Sample Number on the laboratory Form I matches the Sample Identification on the COC. Read the laboratory case narrative for additional information.

1. Any samples received for analysis that were not analyzed shall be noted in the data validation report, along with the reason(s) for failure to analyze the samples, if the reason(s)

can be determined. Conversely, samples that were analyzed for semivolatiles, but were not requested should also be noted.

2. Any discrepancies in sample naming between the COC and sample results form shall be noted in the data validation report with the correct sample name being identified if the correct sample name can be determined.
3. If the receiving laboratory transferred the samples to another laboratory for analysis, both the original COCs and transfer COCs shall be present. Document in the data validation report if the transfer COCs are not present.
4. Internal COC is required for all samples, extracts, and digestates from receipt to disposal. Verify the internal COC forms for completeness. Document in the data validation report if the internal COC forms are not present.
5. Each individual cooler shall have an individual COC that lists only samples contained within that cooler. Document in the data validation report if multiple coolers appear on one COC.

4.1.3 Holding Times

Level C and Level D:

Holding times for semivolatile organics are measured from the time of collection (as shown on the COC) to the time of sample extraction and from the time of sample extraction to the time of sample analysis (as shown on the Form I). Samples and extracts must be stored and refrigerated at above freezing to 6°C until the time of analysis.

Water samples shall be unpreserved and refrigerated at above freezing to 6°C and shall be extracted within 7 days of collection and analyzed within 40 days of extraction.

Soil samples shall be unpreserved and refrigerated at above freezing to 6°C and shall be extracted within 14 days of collection and analyzed within 40 days of extraction.

1. If the holding time is exceeded, flag all associated positive results as estimated "J" and all associated limits of detection (LODs) (nondetects) as estimated "UJ," and document that holding times were exceeded.
2. If holding times are grossly exceeded by greater than a factor of 2.0 (e.g., a non-water sample has a holding time of more than 14 days), detects will be qualified as estimated "J" and nondetects as unusable "R."

4.2 GC/MS Instrument Performance Check (Full Scan)

Level C and Level D:

GC/MS instrument performance checks or tune checks are performed for the Full scan analyses to ensure mass resolution, identification, and to some degree, sensitivity. Instrument performance checks are not required for samples analyzed by selected ion monitoring (SIM). These criteria are not sample specific. Conformance is determined using standard materials; therefore, these criteria should be met in all circumstances.

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance

check, decafluorotriphenylphosphine (DFTPP) for semivolatile analysis, must meet the ion abundance criteria given below.

Table II-C-1: Ion Abundance Criteria – DFTPP (SW-846 8270C)

m/z	Ion Abundance Criteria
51	30.0–60.0% of m/z 198
68	Less than 2.0% of m/z 69
70	Less than 2.0% of m/z 69
127	40.0–60.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base peak, 100% relative abundance
199	5.0–9.0% of m/z 198
275	10.0–30.0% of m/z 198
365	Greater than 1.0% of m/z 198
441	Present, but less than m/z 443
442	Greater than 40.0% of m/z 198
443	17.0–23.0% of m/z 442

% percent
m/z mass-to-charge ratio

Table C-II-2: Ion Abundance Criteria – DFTPP (SW-846 8270D)

m/z	Ion Abundance Criteria
51	10.0–80.0% of m/z 198
68	Less than 2.0% of m/z 69
70	Less than 2.0% of m/z 69
127	10.0–80.0% of m/z 198
197	Less than 2.0% of m/z 198
198	Base peak, 100% relative abundance
199	5.0–9.0% of m/z 198
275	10.0–60.0% of m/z 198
365	Greater than 1.0% of m/z 198
441	Present, but less than 24.0% m/z 442
442	Greater than 50.0% of m/z 198
443	15.0–24.0% of m/z 442

Check that all sample runs are associated with an injection. Make certain that a DFTPP performance check is present for each 12-hour period samples are analyzed (Form V [or equivalent]). Verify that all samples were analyzed within 12 hours of DFTPP injection.

If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. The most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and type of instrumentation; therefore, the critical ion abundance criteria for DFTPP are the mass to charge (m/z) ratios for 198/199 and 442/443. The relative abundances for m/z 68, 70, 197, and 441 are also very important.

The relative abundances of m/z 51, 127, 275, and 365 are of lesser importance. For example, if the relative abundance of m/z 365 is zero, minimum detection limits may be affected. However, if m/z 365 is present, but less than the 1.0 percent minimum abundance criteria, the deficiency is not as serious. Use professional judgment when samples are analyzed beyond the 12-hour time limit.

DFTPP should also be used to assess GC column performance and injection port inertness. Degradation of 4,4'-dichlorodiphenyltrichloroethane to 4,4'-dichlorodiphenyldichloroethane and 4,4'-dichlorodiphenyldichloroethylene should not exceed 20 percent. Benzidine and pentachlorophenol should be present at their normal responses and should not exceed a tailing factor of 2 using the equation presented in EPA SW-846 8270D (or most current version). Decisions to use analytical data associated with DFTPP instrument performance checks not meeting requirements should be noted in the data validation report.

Level D:

Verify by recalculating from the raw data (mass spectral listing) that the mass assignment is correct and that the mass listing is normalized to the specified m/z. If transcription or rounding errors are discovered on the Form V (or equivalent), request a resubmittal from the laboratory. Validate the data using the criteria outlined above.

4.3 CALIBRATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the semivolatile target compound list for both Full Scan and SIM analyses.

4.3.1 Initial Calibration

Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing an acceptable calibration curve for both Full Scan and SIM analyses.

Level C and Level D:

1. Evaluate the average relative response factors (RRFs) for all target compounds by checking Form VI (or equivalent).
2. If any of the semivolatile target compounds listed in Table C-II-3below has an average RRF of less than 0.01 or any other semivolatile target compound has an average RRF of less than 0.05, flag positive results for that compound as estimated "J" and nondetects as unusable "R" in associated samples.

Table C-II-3: Semivolatile Compounds Exhibiting Poor Response

2,2'-Oxybis-(1-chloropropane)	Benzaldehyde
4-Chloroaniline	4-Nitroaniline
Hexachlorobutadiene	4,6-Dinitro-2-methylphenol
Hexachlorocyclopentadiene	N-Nitrosodiphenylamine
2-Nitroaniline	3,3'-Dichlorobenzidine
3-Nitroaniline	1,1'-Biphenyl
2,4-Dinitrophenol	Dimethylphthalate
4-Nitrophenol	Diethylphthalate

Acetophenone	1,2,4,5-Tetrachlorobenzene
Caprolactam	Carbazole
Atrazine	Butylbenzylphthalate
Di-n-butylphthalate	Di-n-octylphthalate
Bis(2-ethylhexyl)phthalate	

3. Check Form VI (or equivalent) and evaluate the percent relative standard deviation (%RSD) for all target compounds. If any semivolatile target compound has a %RSD of greater than 15 percent, flag detects for the affected compounds as "J" and nondetects as "UJ" in the associated samples that correspond to that initial calibration.

Level D:

1. Verify the files reported on Form VI (or equivalent) against the quantitation reports, mass spectra, and chromatograms. If the files do not match, the RRFs reported are likely to be from another initial calibration and will have to be changed. Request a resubmittal from the laboratory.
2. Recalculate the average RRFs and %RSDs reported on Form VI (or equivalent) for one compound per internal standard from the raw data (preferably compounds which were identified in the samples) on the low-point calibration standard and one additional calibration standard. If errors are discovered, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.3.2 Initial Calibration Verification

The initial calibration curve must be verified with a standard that has been purchased or prepared from an independent source each time initial calibration is performed. A standard from the same manufacturer but independently prepared from different source materials may also be used as an independent source. This initial calibration verification (ICV) must contain all of the method target compounds.

Level C and Level D:

1. Verify the ICV was analyzed following the initial calibration and contained all method target compounds.
2. If any target analyte has a percent difference (%D) greater than 20 percent, flag detects for the affected compounds as estimated "J" and nondetects as estimated "UJ" in all samples associated with the initial calibration.

Level D:

Verify from the raw data that there were no calculation or transcription errors by recalculating a percentage of the ICV calculations.

4.3.3 Continuing Calibration

The continuing calibration checks document satisfactory maintenance and adjustment of the instrument on a day-to-day basis for both Full Scan and SIM analyses.

Level C and Level D:

1. Continuing calibration standards containing both target compounds and system monitoring compounds must be analyzed every 12 hours during operation. Evaluate the continuing RRFs on Form VII (or equivalent).
2. Ensure that the average RRFs reported on Form VII (or equivalent) correspond to the average RRFs reported on Form VI (or equivalent) for the corresponding initial calibration.
3. If any of the semivolatile target compounds listed in Table C-II-3 has an average RRF of less than 0.01 or any other semivolatile target compound has an average RRF of less than 0.05, flag positive results for that compound as estimated "J" and nondetects as unusable "R" in associated samples.
4. If any semivolatile target compound has a %D between the initial calibration average RRF and continuing calibration RRFs outside 20 percent, flag all detects as "J" and all nondetects as "UJ" in all associated samples that correspond to that continuing calibration.
5. An ending continuing calibration is required by DoD QSM Appendix B (an ending continuing calibration is not required by the method) and professional judgment should be used in qualifying associated data when the %D is outside 50 percent.

Level D:

1. Verify the file reported on Form VII (or equivalent) against the raw data for the continuing calibration. If the file does not match, the RRFs reported are likely to be from another continuing calibration and will have to be changed. Request a resubmittal from the laboratory.
2. Recalculate the reported RRFs and %Ds reported on Form VII (or equivalent) for one compound per internal standard. If errors are discovered, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.4 BLANKS

Method blank analytical results are assessed to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all associated data must be carefully evaluated to determine whether there is any bias on the data, or if the problem is an isolated occurrence not affecting other data. Results may not be corrected by subtracting any blank values.

Level C and Level D:

1. The reviewer should identify samples associated with each method blank using Form IV (or equivalent). Verify that method blank analysis has been reported per matrix and concentration level for each set of samples. Each sample must have an associated method blank. Qualify positive results in samples with no method blank as unusable "R." Nondetects do not require qualification.
2. Compare the results of each method blank with the associated sample results. The reviewer should note that the blank analyses may not involve the same weights, volumes, percent moistures, or dilution factors as the associated samples. These factors must be taken into consideration when applying the criteria discussed below, such that a comparison of the total amount of contamination is actually made.

3. If a compound is found in the blank, but not in the associated sample, no action is taken.
4. Compounds that are detected in both the sample and the associated blank with the exception of bis(2-ethylhexylphthalate) shall be qualified when the sample concentration is less than the limit of quantitation (LOQ) and the blank concentration is less than, greater than, or equal to the LOQ. Bis(2-ethylhexylphthalate) shall be qualified when the sample concentration is less than five times (5×) the LOQ and the blank concentration is less than, greater than, or equal to 5× LOQ. Care should be taken to factor in the percent moisture when comparing detects in the sample and the method blank. The applicable review qualifier(s) are summarized in Table C-II-4.

Table C-II-4: Blank Qualifications

Sample Result	Sample Value	Reviewer Qualifier(s)
Less than LOQ* and blank result is <, > or = LOQ*	Leave as reported	U
≥ LOQ*, blank result is < LOQ*	Leave as reported	None
≥ LOQ*, blank result is > LOQ* and sample result < blank result	Leave as reported	Use professional judgment
≥ LOQ*, blank result is > LOQ* and sample result ≥ blank result	Leave as reported	Use professional judgment
≥ LOQ* and blank result is = LOQ*	Leave as reported	Use professional judgment

*5x LOQ for bis(2-ethylhexylphthalate)

In the case wherein both the sample concentration and the blank concentration are greater than or equal to the LOQ, previously approved criteria as identified in the project planning documents may be applied to qualify associated sample results. Otherwise, qualify sample results as non-detect "U" when the sample concentration is less than or equal to 10 times the blank concentration (10× rule) for the phthalates listed in Table C-II-5 and tentatively identified compounds (TICs). For all other compounds, qualify sample results as non-detect "U" when the sample concentration is less than or equal to 5× the blank concentration (5× rule).

Table C-II-5: Phthalates

Dimethylphthalate
Diethylphthalate
Di-n-butylphthalate
Butylbenzylphthalate
Bis(2-ethylhexylphthalate)
Di-n-octylphthalate

5. If gross contamination exists in the blanks (i.e., saturated peaks by GC/MS), all compounds affected shall be flagged as unusable "R" due to interference in all samples affected and this shall be noted in the data validation comments.
6. If target compounds other than common laboratory contaminants are found at low levels in the blank(s), it may be indicative of a problem at the laboratory and shall be noted in the data validation report.
7. Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination

introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. It may be impossible to verify this source of contamination; however, if the reviewer determines that the contamination is from a source other than the sample, the data should be qualified. The sample value shall be reported as a nondetect and the reason shall be documented in the data validation report.

Level D:

1. Verify all target compound and TIC detects found in the method blanks against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid or the tentative identity is in error, request for a corrected Form I for the method blank from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.5 Blank Spikes and Laboratory Control Samples

Blank spike/laboratory control sample (LCS) recoveries must be within the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established for a given sample matrix. Use in-house limits if compounds are not listed in Appendix C or project limits are not specified.

Level C and Level D:

1. If the blank spike/LCS results are 0 percent, only the spiked compounds that showed low recovery in all associated samples shall be flagged as unusable "R" for nondetects and estimated "J" for detects.
2. If blank spike/LCS results are below the control limits (but above 0 percent), spiked compounds which showed low recovery in all associated samples shall be flagged as estimated "UJ" or "J."
3. If blank spike/LCS results are above the control limits, detects for only the spiked compounds which showed high recovery in all associated samples shall be flagged as estimated "J."
4. If the laboratory analyzes a blank spike duplicate/LCS duplicate (LCSD), evaluate and qualify the LCSD results using the criteria noted above.
5. If the relative percent difference (RPDs) between LCS and LCSD results are above the control limits (use the MS/MSD RPD control limits identified in DoD QSM Appendix B, if none are available use laboratory in-house limits), spiked compounds which showed high RPD in all associated samples shall be flagged as estimated "UJ" or "J."

Level D:

To check that the spike percent recovery was calculated and reported correctly using the following equation, recalculate one or more spike recoveries per matrix (and any spike that would result in the qualification of a sample).

$$\%\text{Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Quantity determined by analysis

Q_a = Quantity added to samples/blanks

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.6 System Monitoring compounds (Surrogate Spikes)

Laboratory performance on individual samples is established by means of surrogate spiking activities. All samples are spiked with surrogate compounds prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects because of factors such as interferences and high concentrations of compounds. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. The following procedures shall be followed:

Level C and Level D:

1. Sample and blank surrogate recoveries for semivolatiles must be within the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established. Use in-house limits if surrogates are not listed in Appendix C or project limits are not specified. Verify that no samples or blanks have surrogates outside the criteria from Form II (or equivalent).
2. If two or more surrogates in a base/neutral fraction or two or more surrogates in an acid fraction are out of specification, or if at least one surrogate has a recovery of less than 10 percent, then the sample should be re-analyzed though surrogate results still could be outside the criteria. (Note: When unacceptable surrogate recoveries are followed by successful re-analyses, the laboratories are required to report only the successful run unless the re-analyses were performed outside the holding times. Laboratories do not have to perform a re-analysis if a matrix spike/matrix spike duplicate was performed on the sample with out-of-control surrogate results showing the same matrix effects.)
3. The laboratory has failed to perform satisfactorily if surrogate recoveries are out of specification with no evidence of re-analysis. The non-surrogate recoveries shall be documented in the data validation report.

4. If two or more surrogates in the base/neutral fraction or two or more surrogates in the acid fraction are less than lower acceptance limit, but have a recovery greater than or equal to 10 percent, qualify positive results for that fraction as estimated "J" and nondetects as estimated "UJ." (Note that all phenols pertain to the acid fraction; all remaining compounds correspond to the base neutral fraction.)
5. If any surrogate in a fraction shows less than 10 percent recovery, qualify positive results for that fraction as estimated "J," and nondetects for the fraction as unusable "R."
6. If two or more surrogates in either base/neutral or acid-faction have a recovery greater than the upper acceptance limit, detected compounds in that fraction are qualified "J." Nondetects should not be qualified.
7. No qualification with respect to surrogate recovery is placed on data unless at least two surrogates in the semivolatile fraction are out of specification or unless any surrogate has less than 10 percent recovery.
8. In the special case of blank analysis with surrogates out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if the samples in the batch show acceptable surrogate recoveries, the reviewer may determine the blank problem to be an isolated occurrence for which no qualification of the data is required.
9. Surrogates may be reported as "diluted out" (D); if dilution is such that the surrogate can no longer be detected. If this is the case, note in the data validation report that surrogate evaluation could not be performed due to a high dilution factor. A full evaluation of the sample chromatogram and quantitation report may be necessary to determine that surrogates are truly "diluted out."

Level D:

Verify that the surrogate percent recovery was calculated and reported correctly using the following equation. Recalculate all surrogate recoveries for one sample per matrix:

$$\%\text{Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Quantity determined by analysis

Q_a = Quantity added to samples/blanks

If transcription errors are discovered on Form II (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

Matrix Spike/Matrix Spike Duplicate (MS/MSD) data are used to determine the effect of the matrix on a method's recovery efficiency and precision for a specific sample matrix.

No action is taken on MS/MSD data alone to qualify an entire data package. Using informed professional judgment; however, the data reviewer may use the MS/MSD results in conjunction with other QC criteria (i.e., surrogates and LCS) and determine the need for some qualification of the data.

The data reviewer should first try to determine the extent to which the results of the MS/MSD affect the associated data. This determination should be made with regard to the MS/MSD sample itself, as well as specific compounds for all samples associated with the MS/MSD.

In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. It may be determined through the MS/MSD results, however, that a laboratory is having a systematic problem in the analysis of one or more compounds, which affects all associated samples.

Note: If a field blank was used for the MS/MSD, the information must be included in the data validation summary. Sample matrix effects have not been observed with field blanks therefore the recoveries and precision do not reflect the analytical impact of the site matrix.

Level C and Level D:

The laboratory must spike and analyze an MS/MSD from the specific project site as required for each matrix type and analytical batch.

1. MS/MSD data should be reported on a MS/MSD summary form similar to Form III (or equivalent).
2. Compare the percent recovery (%R) and relative percent difference (RPD) for each spiked compound with the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established. Use in-house limits if spiked compounds are not listed in Appendix C or project limits are not specified.
3. If MS/MSD results are 0 percent, only the spiked compounds that showed low recovery in the parent sample shall be flagged as unusable "R" for nondetects and estimated "J" for detects.
4. If MS/MSD results are below the control limits (but above 0 percent), spiked compounds which showed low recovery in the parent sample shall be flagged as estimated "UJ" or "J."
5. If MS/MSD results are above the control limits, detects for only the spiked compounds which showed high recovery in the parent sample shall be flagged as "J."
6. If the RPDs between MS and MSD results are greater than 20 percent, detects for only the spiked compounds which showed high RPD in the parent sample shall be flagged as estimated "J."
7. Failure of MS/MSD due to the presence of a target compound in the parent sample at greater than two times the spike concentration or diluted by more than a factor of two should not result in any qualifications. Note the incident in the data validation report.

Level D:

Check the raw data and recalculate one or more percent recoveries (%Rs) and RPDs, especially %Rs and RPDs that resulted in the qualification of data, using the following equations to verify that results on Form III (or equivalent) are correct.

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

$$RPD = \frac{ABS|SSR - SDR|}{(SSR + SDR)/2} \times 100$$

Where:

SA	=	spike added
SR	=	sample result
SSR	=	spiked sample result
SDR	=	spiked duplicate result
ABS	=	absolute value

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.8 FIELD QC SAMPLES

Field QC samples discussed in this section of the procedures are equipment blanks, field blanks, field duplicates, and field triplicates.

4.8.1 Equipment Blanks and Field Blanks

Compounds detected in equipment blanks indicate the possibility of cross-contamination between samples due to improper equipment decontamination.

A field blank sample may be collected from each source of water used during each sampling event. The field blank may be analyzed to assess whether the chemical nature of the water used in decontamination may have affected the analytical results of site samples.

If semivolatile organic compounds are detected in the equipment blanks and/or field blanks, the procedure for the qualification of associated sample results is identical to the criteria outlined in Section 4.4 of this procedure.

Level C and Level D:

1. Determine which field QC samples apply to samples in the sample delivery group (SDG).

2. Ensure that units are correct when applying field QC blank qualifications. If samples are soil matrix, results must first be converted to microgram per liter from microgram per kilogram (µg/kg) to make correct comparisons.
3. Because of the way in which the field blanks and equipment blanks are sampled, equipment blanks are not qualified because of field blank contamination. The affected samples are qualified, however, by either the field blank or equipment blank results, whichever has the higher contaminant concentration.
4. Equipment blanks and field blanks are only qualified with method blank results in order to account for laboratory contamination.

Level D:

1. Verify all target compound and TIC detects found in the equipment blanks and field blanks against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and verify the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid, or the tentative identity is in error, request for a corrected Form I (or equivalent) for the equipment blank or field blank from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.8.2 Field Duplicates and Field Triplicates

Field duplicates consist of either collocated or subsampled samples. Field duplicates for ground water and surface water samples are generally considered to be collocates. Soil duplicate samples may be homogenized and subsampled in the field (or at the laboratory) to form an original and duplicate sample, or may be an additional volume of sample collected in a separate sample container to form a collocate sample. Field duplicate results are an indication of both field and laboratory precision; the results may be used to evaluate the consistency of sampling practices.

Field triplicates are collected from different, randomly selected locations to verify that an incremental sample truly represents a decision unit. Field triplicate results are more useful than field duplicates to statistically evaluate sampling precision.

Level C and Level D:

1. Check to ensure that field duplicates and/or field triplicates were collected and analyzed as specified in the project planning documents. If the sampling frequency is less than the frequency stated in the planning documents, no qualification of the associated sample results is necessary but the incident shall be discussed in the data validation report.
2. For field duplicate results, if the RPDs are greater than 50 percent for water or 100 percent for soil or as stated in the planning document if more conservative, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.

3. For field triplicate results, if the RSDs are greater than the QC limits stated in the planning document, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.

Level D:

1. Verify all target compound and TIC detects found in the field duplicates and/or field triplicates against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid, or the tentative identity is in error, request for a corrected Form I (or equivalent) for the sample or field duplicate from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.9 INTERNAL STANDARDS PERFORMANCE

Internal standards performance criteria ensure that GC/MS sensitivity and response are stable during every analytical run.

Level C and Level D:

1. If an internal standards area count for a sample is outside –50 percent or +100 percent of the area the initial calibration midpoint standard:
2. Positive results for compounds quantitated using an internal standards area count greater than 100 percent should be qualified as estimated "J." Nondetected compounds should not be qualified.
3. Compounds quantitated using an internal standards area count less than 50 percent should be qualified as estimated "J" for detects and estimated "UJ" for nondetects.
4. If extremely low area counts are reported (less than 20 percent of the area for associated standards), detected compounds should be qualified as estimated "J" and nondetected target compounds should then be qualified as unusable "R."
5. If an internal standards retention time varies by more than 10 seconds from the retention time of the initial calibration midpoint standard, the nondetected target compounds should be qualified as unusable "R" for Level C validation. A Level D validation examination of the raw data should be recommended to the CTO Manager. The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Positive results should be qualified as "NJ" if the mass spectral criteria are met.

Level D:

Verify the internal standard areas reported on Form VIII (or equivalent) from the raw data for at least one sample per SDG, and verify internal standard areas for samples that were qualified due to out-of-control

internal standard areas. If errors are discovered between the raw data and the Form VIII (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.10 TARGET COMPOUND IDENTIFICATION

The objective of the criteria for GC/MS qualitative analysis is to minimize the number of erroneous identifications of target compounds. An erroneous identification can either be false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

The identification criteria can be applied more easily in detecting false positives than false negatives. More information is available for false positives because of the requirement for submittal of data supporting positive identifications. However, negatives, or nondetected compounds, represent an absence of data and are, therefore, more difficult to assess. One example of detecting false negatives is the not reporting of a target compound that is reported as a TIC.

Level C:

Target compound identification is not evaluated for Level C validation because it requires the interpretation of mass spectral raw data.

Level D:

The following criteria should be followed when evaluating raw data.

1. The relative retention times (RRTs) must be within ±0.06 RRT units of the standard RRT.
2. Mass spectra of the sample compound and a current laboratory-generated standard (i.e., the mass spectrum from the associated calibration standard) must match according to the following criteria:
3. All ions present in the standard mass spectrum at a relative intensity greater than 10 percent must be present in the sample spectrum.
4. The relative intensities of these ions must agree within ± 20 percent between the standard and sample spectra. (Example: For an ion with an abundance of 50 percent in the standard spectrum, the corresponding sample ion abundance must be between 30 percent and 70 percent.)
5. Ions present at greater than 10 percent in the sample mass spectrum, but not present in the standard spectrum, must be considered and accounted for.
6. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory and CTO Manager. If it is determined that incorrect identifications were made, all such data should be qualified as not detected "U" or unusable "R."
7. Professional judgment must be used to qualify the data if it is determined that cross-contamination has occurred. Any changes made to the reported compounds or concerns regarding target compound identifications should be clearly indicated in the data validation report.

4.11 COMPOUND QUANTITATION AND REPORTING LIMITS

The objective is to ensure that the reported quantitation results and reporting limits (i.e., LOQ, LOD, detection limit [DL]) are accurate. All soil sample results are reported on a dry weight basis.

Level C and Level D:

1. Verify that the reporting limits for nondetects are equal to the LOD. Verify that an annual DL study was performed or quarterly LOD/LOQ verification checks were performed in accordance with the DoD QSM. The LOD/LOQ verification check must be evaluated to determine whether the laboratory can reliably detect and identify all target analytes at a spike concentration of approximately two times but not more than four times the current reported DL. Qualify nondetects as unusable "R."
2. Check that reported nondetects and positive values have been adjusted to reflect sample dilutions (including clean-up) and for soil samples, sample moisture. When a sample is analyzed at more than one dilution, the lowest LODs are used unless a QC criterion has been exceeded. In this case, the higher LODs from the diluted analysis are used. The least technically sound data will be flagged "R" with a qualification code "D."
3. Verify that LOQs/LODs for soils and sediments were calculated based on dry weight. If the LOQs/LODs were reported based on wet weight, the percent moisture must be factored in and the LOQs/LODs must be adjusted accordingly.
4. Verify that no results exceed the highest calibration standard without being diluted. If a result has exceeded the highest calibration standard, verify that a dilution was performed. If not, qualify the detected compound that required dilution as "J" and document the event in the data validation report.

Level D:

The compound quantitation must be evaluated for all detects by evaluating the raw data. Compound concentrations must be calculated based on the internal standards associated with that compound, as listed in the following equation. Quantitation must be based on the quantitation ion (m/z) specified in the method or project planning document for both the internal standards and target compounds. The compound quantitation must be based on the RRF from the appropriate initial calibration standard.

Water

$$\mu g/L = \frac{A_x \times I_s \times D_f \times V_t}{A_{is} \times ARRF \times V_o \times V_i}$$

Where:

A_x	=	area of characteristic ion (extracted ion current profile) for compound being measured
A_{is}	=	area of characteristic ion for the internal standard
I_s	=	amount of internal standard added (nanograms)
ARRF	=	average relative response factor for compound being measured

V_o = volume of water extracted (milliliter)

D_f = dilution factor

V_t = volume of extract injected (microliter [µL])

V_i = volume of concentrated extract (µL)

Soil/Sediment

Concentration *µg/kg* (Dry weight basis) = $$\frac{A_x \times I_s \times D_f \times V_t \times 2.0_t}{A_{is} \times ARRF \times Ws \times D \times V_i}$$

Where:

A_x, I_s, RRF, A_{is}, V_i, V_t are as given for water, above.

D = $\frac{100 - \%\ moisture}{100}$

W_s = Weight of sample extracted, in grams (g)

The factor of 2.0 in the numerator is used to account for the amount of extract that is not recovered from gel permeation chromatography clean up.

If discrepancies are discovered in the quantitation, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.12 TENTATIVELY IDENTIFIED COMPOUNDS

For each sample analyzed by Full Scan, the laboratory may conduct a mass spectral search of the spectral library and report the possible identity for up to 30 largest semivolatile fraction peaks which are not system monitoring compounds (surrogates), internal standards, or target compounds, but which have area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I SV-TIC [or equivalent]). TICs are not reported for SIM analysis.

Level C and Level D:

1. All TIC results should be qualified "NJ," tentatively identified with approximated concentrations.
2. The reviewer should be aware of common laboratory artifacts and their sources such as siloxane compounds, which indicate capillary column degradation, and carbon dioxide, which indicates a possible air leak in the system. These may be qualified as unusable "R."
3. If a target compound is identified as a TIC by non-target library search procedures, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion.
4. TIC results that are not above the 10× level in the blank should be qualified as unusable, "R." (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)

5. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).

Level D:

1. Check each TIC for each sample using the following criteria.
2. Major ions (greater than 10 percent relative intensity) in the reference spectrum should be present in the sample spectrum.
3. The relative intensities of the major ions should agree within ±20 percent between the sample and the reference spectra.
4. Molecular ions present in the reference spectrum should be present in the sample spectrum.
5. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference, or co-elution of additional TIC or target compounds.
6. When the above criteria are not met, but in the technical judgment of the data reviewer or mass spectral interpretation specialist, the identification is correct, the data validator may report the identification.
7. Since TIC library searches often yield several candidate compounds having a close matching score, all reasonable choices must be considered. The reviewer may use judgment to change the reported tentative identity.

5. Records

A Form I that has been validated and verified, and has been determined by the data validator to accurately represent the appropriate sample results to be utilized, shall be stamped "NAVFAC PACIFIC VALIDATED." Additionally, sample result forms for which the data has been validated at the Level D validation level shall be stamped or noted "Level D."

Copies of all documents generated by the data validation personnel will be stored for no less than 10 years. The original validated laboratory data shall be archived to the Federal Records Center at project completion.

6. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www2.epa.gov/sites/production/files/documents/qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Environmental Protection Agency, United States (EPA). 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Final Update IV. Office of Solid Waste. Updates available: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

Procedure II-A, *Data Validation.*

7. Attachments

None.

Level C and Level D Data Validation for Total Petroleum Hydrocarbons by SW-846 8015

1. Purpose

This data validation procedure sets forth the standard operating procedure for performance of Level C and Level D data validation of total petroleum hydrocarbons (TPH) data obtained under the United States (U.S.) Navy Environmental Restoration (ER) Program for Naval Facilities Engineering Command (NAVFAC), Pacific and is consistent with protocol in the *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories* (DoD QSM) (DoD 2013). Level B validation is addressed separately in Procedure II-A, *Data Validation*.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Responsibilities

The CTO Manager, the QA Manager or Technical Director, and the CTO QA Coordinator are responsible for ensuring that this procedure is implemented by data validation personnel.

Data validation personnel are responsible for implementing this procedure for validation of all gas chromatography (GC) TPH data.

4. Procedure

This procedure addresses the validation of TPH data obtained using U.S. Environmental Protection Agency (EPA) Method Solid Waste (SW)-846 8015 (EPA 2007). The quality control (QC) criteria identified in this procedure are those specified in the analytical method and the DoD QSM (DoD 2013). Where project specific criteria are identified in the CTO work plan, they will supersede the QC criteria identified in this procedure.

- Form I: Sample Results Summary Form
- Form II: Surrogate Recovery Summary Form
- Form III: Matrix Spike/Matrix Spike Duplicate or Blank Spike/Blank Spike Duplicate Recovery Summary Form

- Form IV: Method Blank Summary Form
- Form VI: Initial Calibration Summary Form
- Form VII: Continuing Calibration Summary Form
- Form VIII: TPH Analytical Sequence Form

Level C data validation consists of review of summary forms only while Level D data validation requires review of both summary forms and all associated raw data. Data review guidelines and how they apply to the different validation levels are indicated in the following text.

4.1 SAMPLE MANAGEMENT

QA/QC criteria included under sample management are sample preservation, handling, and transport; chain of custody (COC); and holding times.

4.1.1 Sample Preservation, Handling, and Transport

Level C and Level D:

Evaluate sample collection, handling, transport, and laboratory receipt from COC and laboratory receipt checklists to ensure that the samples have been properly preserved and handled.

TPH as Gasoline

1. Water samples must be preserved with hydrochloric acid at or below a pH of 2 and refrigerated at above freezing to 6 degrees Celsius (°C).
2. Soil samples collected in volatile organic analysis (VOA) vials or coring devices must be refrigerated at above freezing to 6°C. If the samples are to be analyzed after the 48-hour holding time, the laboratory must preserve the samples with sodium bisulfate or methanol or freeze upon receipt in accordance with EPA SW-846 Method 5035 (EPA 2007).
3. If the analyzed aqueous VOA vial contains air bubbles or headspace, is cracked, or has a cracked cap, positive values shall be flagged as estimated "J" and nondetects as estimated "UJ." The sample data may be qualified as unusable "R" if the container damage is extensive or improper sealing is identified.
4. VOA vials are to be shipped in coolers that are maintained at above freezing to 6°C. If the temperature exceeds 6°C, but is less than or equal to 10°C, note this in the data validation report. If the temperature of receipt is greater than or equal to 11°C, positive values shall be flagged as estimated "J" and nondetects as estimated "UJ." If the temperature of receipt is greater than or equal to 15°C, positive values shall be flagged as estimated "J" and nondetects as unusable "R." If the temperature is below 0°C, special note should be made that the samples were frozen and no qualification shall be required. In the event that both a cooler temperature and a temperature blank were measured, the temperature blank shall be evaluated for temperature compliance as it best assimilates the condition of the samples; however, both temperatures shall be noted in the data validation report.

TPH as Extractables

1. Samples are to be shipped in coolers that are maintained at above freezing to 6°C. If the temperature exceeds 6°C but is less than or equal to 10°C, note this in the data validation

report. If the temperature of receipt is greater than or equal to 11°C, positive values shall be flagged as estimated "J" and nondetects as estimated "UJ." If the temperature is below 0°C, special note should be made that the samples were frozen and no qualification shall be required. In the event that both a cooler temperature and a temperature blank were measured, the temperature blank shall be evaluated for temperature compliance as it best assimilates the condition of the samples; however, both temperatures shall be noted in the data validation report.

2. Water samples shall not be preserved; they shall only be kept cool. If the water samples were inappropriately preserved with acid, the samples should not be analyzed. Analysis of an inappropriately preserved sample by the laboratory may require that all results be reported as unusable "R."
3. If the temperature of the cooler upon receipt at the laboratory was not recorded, document that the laboratory is noncompliant.

If the receiving laboratory transferred the samples to another laboratory for analysis, apply the same temperature criteria to both the transfer COC and the original COC.

4.1.2 Chain of Custody

Level C and Level D:

Examine the COC for legibility and check that all TPH analyses requested on the COC have been performed by the laboratory. Ensure that the COC Sample Number on the laboratory Form I (or equivalent) matches the Sample Identification on the COC. Read the laboratory case narrative for additional information.

1. Any samples received for analysis that were not analyzed shall be noted in the data validation report, along with the reason(s) for failure to analyze the samples, if the reason(s) can be determined. Conversely, samples that were analyzed for TPH but were not requested should also be noted.
2. Any discrepancies in sample naming between the COC and Form I (or equivalent) shall be noted in the data validation report with the correct sample name being identified if the correct sample name can be determined.
3. If the receiving laboratory transferred the samples to another laboratory for analysis, both the original COCs and transfer COCs shall be present. Document in the data validation report if the transfer COCs are not present.
4. Internal COC is required for all samples, extracts, and digestates from receipt to disposal. Verify the internal COC forms for completeness. Document in the data validation report if the internal COC forms are not present.
5. Each individual cooler shall have an individual COC that lists only samples contained within that cooler. Document in the data validation report if multiple coolers appear on one COC.

4.1.3 Holding Times

Level C and Level D:

Holding times for TPH are measured from the time of collection (as shown on the COC) to the time of sample extraction and from the time of sample extraction to the time of sample analysis (as shown

on the Form I [or equivalent]). Samples and extracts must be stored and refrigerated at above freezing to 6°C until the time of analysis.

TPH as Gasoline

1. Water samples must be preserved with hydrochloric acid and refrigerated at above freezing to 6°C. Preserved water samples shall be analyzed within 14 days from the collection date. If there is no indication of chemical preservation, assume samples are unpreserved. For unpreserved water samples, the holding time is 7 days from date collected.
2. Soil samples collected in VOA vials or coring devices that are unpreserved must be refrigerated at above freezing to 6°C and analyzed within 48 hours from the collection date. Soil samples that are preserved with sodium bisulfate or methanol, or frozen upon laboratory receipt shall be analyzed within 14 days from the collection date.

TPH as Extractables

Water samples shall be unpreserved and refrigerated at above freezing to 6°C and shall be extracted within 7 days of collection and analyzed within 40 days of extraction.

Soil samples shall be unpreserved and refrigerated at above freezing to 6°C and shall be extracted within 14 days of collection and analyzed within 40 days of extraction.

1. If the holding time is exceeded, flag all associated positive results as estimated "J" and all associated limits of detection (LODs) (nondetects) as estimated "UJ," and document that holding times were exceeded.
2. If holding times are grossly exceeded by greater than a factor of 2.0 (e.g., a non-preserved water sample has an extraction holding time of more than 14 days), detects will be qualified as estimated "J" and nondetects as unusable "R."

4.2 GC Instrument Performance

Level C:

Instrument performance is not evaluated for Level C validation.

Level D:

Evaluate the blank, standard, laboratory control sample, and sample chromatograms to ascertain the performance of the chromatographic system. Professional judgment should be used to qualify the data when unacceptable chromatographic conditions preclude proper quantitation or identification of TPH.

4.3 Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that an instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that an instrument is capable of acceptable performance at the beginning of a sequence, and continuing calibration checks document satisfactory maintenance and adjustment of the instrument on a day-to-day basis.

Level C and Level D:

1. The proper analytical sequence must be followed to ensure proper quantitation and identification of all target compounds. For the quantitation analysis, standards containing all target compounds, (specific hydrocarbon products or n-alkanes) must be analyzed in the initial calibration at the beginning of the sequence. If n-alkane ranges rather than specific hydrocarbon products are being reported, n-alkane standards must be run in the initial calibration and should be analyzed periodically to ensure proper identification of the n-alkane range reported. An initial calibration verification standard must be analyzed following each initial calibration. The mid-level standard of the initial calibration must be analyzed after every 10 samples as the continuing calibration and at the end of the sequence to ensure system performance has not degraded. If the proper sequence has not been analyzed, use professional judgment to assess the reliability of the data.
2. The laboratory should report retention time window data for each compound and each column used to analyze the samples. The retention time windows are used for qualitative identification. The laboratory should also report quantitation ranges used for integration when analyzing samples. If the compounds in the continuing calibration standard do not fall within the retention time windows established in the initial calibration, the associated sample results should be carefully evaluated, especially the retention time of the surrogate spike compound. All samples injected after the last in-control standard are potentially affected.

4.3.1 Initial Calibration

Level C and Level D:

For the initial calibration (at least five-points), the relative standard deviation (RSD) of the calibration factor (CF) for each target compound must be less than or equal to 20 percent. Verify the RSDs from the initial calibration summary forms. Alternatively, a linear curve may be used with a coefficient of determination; r^2 equal to or greater than 0.990. A second order calibration curve may also be used after evaluating the laboratory's acceptance criteria. If the initial calibration criteria are not met, flag all associated quantitative results as estimated "J" for detects and estimated "UJ" for nondetects.

Level D:

Verify the percent RSDs, r^2, or laboratory established measure of linearity for the initial calibration from the raw data. Verify the CF for each target compound from the raw data on the low-point calibration standard and one additional calibration standard. If errors are discovered, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.3.2 Initial Calibration Verification

The initial calibration curve must be verified with a standard that has been purchased or prepared from an independent source each time initial calibration is performed. A standard from the same manufacturer but independently prepared from different source materials may also be used as an independent source. This initial calibration verification (ICV) must contain all of the method target compounds.

Level C and Level D:

1. Verify the ICV was analyzed following the initial calibration and contained all method target compounds.
2. If any target analyte has a percent difference (%D) greater than 20 percent, flag detects for the affected compounds as estimated "J" and nondetects as estimated "UJ" in all samples associated with the initial calibration.

Level D:

Verify from the raw data that there were no calculation or transcription errors by recalculating a percentage of the ICV calculations.

4.3.3 Continuing Calibration

Level C and Level D:

Verify the %D from the continuing calibration summary forms. For the continuing calibration, the %D between the CF from the continuing calibration and the average CF from the initial calibration must be less than 20 percent. Alternatively, if a linear (first-order) calibration curve is utilized in the initial calibration, the %D of the calculated amount and the true amount for each compound must be less than or equal to 20 percent. If the continuing calibration criteria are not met, qualify all associated results as estimated "J" for detects and "UJ" for nondetects.

Level D:

Verify the %Ds from the raw data.

4.4 BLANKS

Method blank analytical results are assessed to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all associated data must be carefully evaluated to determine whether there is any bias associated with the data, or if the problem is an isolated occurrence not affecting other data. No contaminants should be present in the method blank(s). The method blank should be analyzed on each GC system used to analyze site samples.

1. The reviewer should identify samples associated with each method blank using Form IV (or equivalent). Verify that method blank analysis has been reported per matrix and concentration level for each set of samples. Each sample must have an associated method blank. Qualify positive results in samples with no method blank as unusable "R." Nondetects do not require qualification.
2. If the method blank was not analyzed on a GC used to analyze site samples, note the deficiency in the data validation report. Professional judgment shall be used for subsequent qualification of the data.
3. Compare the results of each method blank with the associated sample results. The reviewer should note that the blank analyses may not involve the same weights, volumes, percent moistures, or dilution factors as the associated samples. These factors must be taken into consideration when applying the criteria discussed below, such that a comparison of the total amount of contamination is actually made.

4. If a compound is found in the blank, but not in the associated sample, no action is taken.
5. Any compound detected in both the sample and the associated blank shall be qualified when the sample concentration is less than the limit of quantitation (LOQ) and the blank concentration is less than, greater than, or equal to the LOQ. Care should be taken to factor in the percent moisture when comparing detects in the sample and the method blank. The applicable review qualifier(s) are summarized in Table II-H-1.

Table II-H-1: Blank Qualifications

Sample Result	Sample Value	Reviewer Qualifier(s)
Less than LOQ and blank result is <, > or = LOQ	Leave as reported	U
≥LOQ, blank result is <LOQ	Leave as reported	None
≥LOQ, blank result is >LOQ and sample result <blank result	Leave as reported	Use professional judgment
≥LOQ, blank result is >LOQ and sample result ≥blank result	Leave as reported	Use professional judgment
≥LOQ and blank result is = LOQ	Leave as reported	Use professional judgment

6. In the case wherein both the sample concentration and the blank concentration are greater than or equal to the LOQ, previously approved criteria as identified in the project planning documents may be applied to qualify associated sample results. Otherwise, qualify sample results as non-detect "U" when the sample concentration is less than or equal to 5 times the blank concentration (5× rule).
7. Instances of contamination can be attributable to the dilution process. These occurrences are difficult to determine; however, the reviewers should qualify the sample data as nondetects, "U," when the reviewer determines the contamination to be from a source other than the sample.
8. In the event of gross contamination (i.e., saturated peaks) in the blanks, the associated samples must be evaluated for gross contamination. If gross contamination exists in the samples, the affected compounds should be qualified as unusable, "R."

Level D:

1. Verify from the preparation log that the information recorded on Form IV (or equivalent) is correct.
2. Review the results of all blank raw data and Form I (or equivalent) to ensure that there were no false negatives or false positives.
3. Verify all target compound detects found in the method blanks against the raw data. Follow the guidelines specified in Sections 4.9 and 4.10 of this procedure. After the validity of the target compounds are verified, validate the corresponding data using the criteria outlined above for Level C and Level D validation.

4.5 Blank Spikes and Laboratory Control Samples

Blank spike/laboratory control sample (LCS) recoveries must be within the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established for a given sample matrix. Use in-house limits if compounds are not listed in Appendix C or project limits are not specified.

Level C and Level D:

1. If the blank spike/LCS results are 0 percent, only the spiked compounds that showed low recovery in all associated samples shall be flagged as unusable "R" for nondetects and estimated "J" for detects.
2. If blank spike/LCS results are below the control limits (but above 0 percent), spiked compounds which showed low recovery in all associated samples shall be flagged as estimated "UJ" or "J."
3. If blank spike/LCS results are above the control limits, detects for only the spiked compounds which showed high recovery in all associated samples shall be flagged as estimated "J."
4. If the laboratory analyzes a blank spike duplicate/LCS duplicate (LCSD), evaluate and qualify the LCSD results using the criteria noted above.
5. If the relative percent differences (RPDs) between LCS and LCSD results are above the control limits (use the matrix spike [MS]/matrix spike duplicate [MSD] RPD control limits identified in DoD QSM Appendix B, if none are available use laboratory in-house limits), spiked compounds which showed high RPD in all associated samples shall be flagged as estimated "UJ" or "J."

Level D:

To verify that the spike percent recovery was calculated and reported correctly using the following equation, recalculate one spike recovery per matrix (and any spike that would result in the qualification of a sample).

$$\%\text{Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Quantity determined by analysis

Q_a = Quantity added to samples/blanks

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.6 Surrogate Recovery

Laboratory performance on individual samples is established by means of surrogate spiking activities. All samples are spiked with surrogate compounds prior to sample preparation. The

evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects because of factors such as interferences and high concentrations of compounds. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. The following procedures shall be followed:

Level C and Level D:

Sample and blank surrogate recoveries for TPH must be within the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established. Use in-house limits if surrogates are not listed in Appendix C or project limits are not specified. Verify that no samples or blanks have surrogates outside the criteria from Form II (or equivalent).

1. If recovery is below the QC limits for any of the surrogates, but above or equal to 10 percent, flag associated positive results as estimated "J" and nondetects as "UJ."
2. If any surrogate recovery is less than 10 percent, flag all nondetects as unusable "R" and detects as estimated "J." No qualification is applied if surrogates are diluted beyond detection but note in the data validation report that surrogate evaluation could not be performed due to the high dilution factor.
3. If any surrogate recovery is above the upper QC limit, flag associated positive results as estimated "J." No qualification of nondetects is necessary in the case of high recoveries.
4. Surrogates may be reported as "diluted out" (D); if dilution is such that the surrogate can no longer be detected. If this is the case, note in the data validation report that surrogate evaluation could not be performed due to a high dilution factor. A full evaluation of the sample chromatogram may be necessary to determine that surrogates are truly "diluted out."

Level D:

The reported surrogate recoveries on Form II should be verified from the raw data for a representative number of samples.

4.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD data are used to determine the effect of the matrix on a method's recovery efficiency and precision for a specific sample matrix.

No action is taken on MS/MSD data alone to qualify an entire data package. Using informed professional judgment; however, the data reviewer may use the MS/MSD results in conjunction with other QC criteria (i.e., surrogates and LCS) and determine the need for some qualification of the data.

The data reviewer should first try to determine the extent to which the results of the MS/MSD affect the associated data. This determination should be made with regard to the MS/MSD sample itself, as well as specific compounds for all samples associated with the MS/MSD.

In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. It may be determined through the

MS/MSD results, however, that a laboratory is having a systematic problem in the analysis of one or more compounds, which affects all associated samples.

Note: If a field blank was used for the MS/MSD, the information must be included in the data validation summary. Sample matrix effects have not been observed with field blanks therefore the recoveries and precision do not reflect the analytical impact of the site matrix.

Level C and Level D:

The laboratory must spike and analyze a MS/MSD from the specific project site as required for each matrix type and analytical batch.

1. MS/MSD data should be reported on a MS/MSD summary form similar to Form III (or equivalent).
2. Compare the percent recovery (%R) and RPD for each spiked compound with the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established. Use in-house limits if spiked compounds are not listed in Appendix C or project limits are not specified.
3. If MS/MSD results are 0 percent, only the spiked compounds that showed low recovery in the parent sample shall be flagged as unusable "R" for nondetects and estimated "J" for detects.
4. If MS/MSD results are below the control limits (but above 0 percent), spiked compounds which showed low recovery in the parent sample shall be flagged as estimated "UJ" or "J."
5. If MS/MSD results are above the control limits, detects for only the spiked compounds which showed high recovery in the parent sample shall be flagged as "J."
6. If the RPDs between MS and MSD results are greater than 30 percent, detects for only the spiked compounds which showed high RPD in the parent sample shall be flagged as estimated "J."
7. Failure of MS/MSD due to the presence of a target compound in the parent sample at greater than 2 times the spike concentration and or diluted by more than a factor of 2 should not result in any qualifications. Note the incident in the data validation report.

Level D:

Check the raw data and recalculate one or more %Rs and RPDs, especially %Rs and RPDs that resulted in the qualification of data, using the following equations to verify that results on Form III (or equivalent) are correct.

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

$$RPD = \frac{ABS|SSR - SDR|}{(SSR + SDR)/2} \times 100$$

Where:

SA	=	spike added
SR	=	sample result
SSR	=	spiked sample result
SDR	=	spiked duplicate result
ABS	=	absolute value

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.8 FIELD QC SAMPLES

Field QC samples discussed in this section of the procedures are equipment blanks, field blanks, field duplicates, and field triplicates.

4.8.1 Equipment Blanks and Field Blanks

Compounds detected in equipment blanks indicate the possibility of cross-contamination between samples due to improper equipment decontamination.

A field blank sample may be collected from each source of water used during each sampling event. The field blank may be analyzed to assess whether the chemical nature of the water used in decontamination may have affected the analytical results of site samples.

If TPH compounds are detected in the equipment blanks and/or field blanks, the procedure for the qualification of associated sample results is identical to the criteria outlined in Section 4.4 of this procedure.

Level C and Level D:

1. Determine which field QC samples apply to samples in the sample deliver group.
2. Ensure that units are correct when applying field QC blank qualifications. If samples are soil matrix, results must first be converted to micrograms per liter from micrograms per kilogram to make correct comparisons.
3. Because of the way in which the field blanks and equipment blanks are sampled, equipment blanks are not qualified because of field blank contamination. The affected samples are qualified, however, by either the field blank or equipment blank results, whichever has the higher contaminant concentration.
4. Equipment blanks and field blanks are only qualified with method blank results in order to account for laboratory contamination.

Level D:

Compound identification and quantification of field blank and equipment blank samples must be verified. Follow the guidelines specified in Sections 4.9 and 4.10 of this procedure.

4.8.2 Field Duplicates and Field Triplicates

Field duplicates consist of either collocated or subsampled samples. Field duplicates for ground water and surface water samples are generally considered to be collocates. Soil duplicate samples may be homogenized and subsampled in the field (or at the laboratory) to form an original and duplicate sample, or may be an additional volume of sample collected in a separate sample container to form a collocate sample. Field duplicate results are an indication of both field and laboratory precision; the results may be used to evaluate the consistency of sampling practices.

Field triplicates are collected from different, randomly selected locations to verify that an incremental sample truly represents a decision unit. Field triplicate results are more useful than field duplicates to statistically evaluate sampling precision.

Level C and Level D:

1. Check to ensure that field duplicates were collected and analyzed as specified in the project planning documents. If the sampling frequency is less than the frequency stated in the planning documents, no qualification of the associated sample results is necessary but the incident shall be discussed in the data validation report.
2. For field duplicate results, if the RPDs are greater than 50 percent for water or 100 percent for soil or as stated in the planning document if more conservative, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.
3. For field triplicate results, if the RSDs are greater than the QC limits stated in the planning document, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.

Level D:

Before comparison of duplicates and/or triplicates, the compound identification and quantification must be verified. Follow the guidelines specified in Sections 4.9 and 4.10 of this procedure.

4.9 TARGET COMPOUND IDENTIFICATION

Qualitative criteria for compound identification have been established to minimize the number of erroneous identifications of compounds. An erroneous identification can be either a false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

Level C:

Compound identification is not verified for Level C validation.

Level D:

1. Review Form I or equivalent. Check for errors.

2. Verify that the retention times of sample compounds reported on the Form X or equivalent fall within the calculated retention time windows.
3. Evaluate all sample chromatograms to ensure that the TPH results were properly identified. Presence of unknown single peaks may result in false positives or false negatives. The reviewer should use professional judgment in evaluating the effect of interference.

4.10 COMPOUND QUANTITATION AND REPORTING LIMITS

The objective is to ensure that the reported quantitation results and reporting limits (i.e., LOQ, LOD, detection limit [DL]) are accurate. All soil sample results are reported on a dry weight basis.

Level C:

Specific compound quantitation is not verified for Level C validation.

Level C and Level D:

1. Verify that the reporting limits for nondetects are equal to the LODs. Verify that an annual DL study was performed or quarterly LOD/LOQ verification checks were performed in accordance with the DoD QSM. The LOD/LOQ verification check must be evaluated to determine whether the laboratory can reliably detect and identify all target analytes at a spike concentration of approximately two times but not more than four times the current reported DL. Qualify nondetects as unusable "R."
2. Check that reported nondetects and positive values have been adjusted to reflect sample dilutions and for soil samples, sample moisture. When a sample is analyzed at more than one dilution, the lowest LODs are used unless a QC criterion has been exceeded. In this case, the higher LODs from the diluted analysis are used. The least technically sound data will be flagged "R" with a qualification code "D."
3. Verify that reported limits for soils and sediments were calculated based on dry weight. If the LOQs/LODs were reported based on wet weight, the percent moisture must be factored in and the LOQs/LODs must be adjusted accordingly.
4. If a sample requiring a dilution analysis due to a target compound detect exceeding the calibration linear range was not re-analyzed at a dilution, the compound exceeding calibration range shall be qualified as estimated "J."
5. If the laboratory re-analyzed a sample and submitted both sample results, the reviewer must determine which of the two analyses has better data quality. Only one analysis should be reported and the other is rejected.

Level D:

1. Compound quantification should be verified by recalculation from the raw data for a representative number of samples.
2. Verify from the standard chromatograms that the instrument sensitivity is adequate to support the LODs. Poor sensitivity may result in elevated LODs.

5. Records

A Form I that has been validated and verified, and has been determined by the data validator to accurately represent the appropriate sample results to be utilized, shall be stamped "NAVFAC PACIFIC VALIDATED." Additionally, sample result forms for which the data has been validated at the Level D validation level shall be stamped or noted "Level D."

Copies of all documents generated by the data validation personnel will be stored for no less than 10 years. The original validated laboratory data shall be archived to the Federal Records Center at project completion.

6. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www2.epa.gov/sites/production/files/documents/qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories*. Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Environmental Protection Agency, United States (EPA). 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*. 3rd ed., Final Update IV. Office of Solid Waste. Updates available: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

Procedure II-A, *Data Validation*.

7. Attachments

None.

Level C and Level D Data Validation for Wet Chemistry Analyses

1. Purpose

This data validation procedure sets forth the standard operating procedure for performance of Level C and Level D data validation of wet chemistry parameters data obtained under the United States (U.S.) Navy Environmental Restoration (ER) Program for Naval Facilities Engineering Command (NAVFAC), Pacific and is consistent with protocol in the *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories* (DoD QSM) (DoD 2013). Cursory validation is addressed separately in Procedure II-A, *Data Validation*.

It covers the following parameters:

- Alkalinity (Standard Methods for the Examination of Water and Wastewater Method 2320B and U.S. Environmental Protection Agency [EPA] Method 310.1 [EPA 2007])
- Bromide, Chloride, Fluoride, Nitrate, Nitrite, Orthophosphate, and Sulfate (EPA Method Solid Waste [SW]-846 9056 and EPA Method 300.0)
- Chemical oxygen demand (EPA Method 410)
- Chloride (EPA Method 325.3)
- Chromium VI (EPA Method SW-846 7195/7196A/7197/7198/7199 and EPA method 218.6 and 218.7)
- Cyanide (EPA Method SW-846 9010B/9012A and EPA Method 335)
- Fluoride (EPA Method 340.2)
- Surfactants (M.B.A.S.) (EPA Method 425.1)
- Nitrate/Nitrite (EPA Method 353.2 and 353.3)
- Perchlorate (EPA Method 314.0)
- Phosphate (EPA Method 365.3)
- Sulfate (EPA Methods 375.3 and 375.4)
- Sulfide (EPA Method 376.1)
- Total dissolved solids (EPA Method 160.1)
- Total suspended solids (EPA Method 160.2)
- Total organic carbon (EPA Method SW-846 9060, Lloyd Kahn, and Walkley-Black)
- Total organic halides (EPA Method SW-846 Method 9020)
- Total recoverable petroleum hydrocarbons (EPA Method 418.1)
- pH (EPA Method SW-846 Method 9040 and EPA Method 150.1)
- Total hardness (Standard Methods for the Examination of Water and Wastewater Method 314A and EPA Method 130.1)

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Responsibilities

The CTO Manager, the QA Manager or Technical Director, and the CTO QA Coordinator are responsible for ensuring that this procedure is implemented by data validation personnel.

Data validation personnel are responsible for implementing this procedure for validation of all wet chemistry data.

4. Procedures

This procedure addresses the validation of wet chemistry parameters data obtained using EPA Method SW-846 7195/7196A/7197/7198/7199/9000, *Standard Methods for the Examination of Water and Wastewater*, 21st Edition, 2005, (APHA 2005) and EPA *Methods for Chemical Analysis of Water and Wastes*, revised March 1983 (EPA 1983). The quality control (QC) criteria identified in this procedure are those specified in the analytical method and the DoD QSM (DoD 2013). Where project specific criteria are identified in the CTO work plan, they will supersede the QC criteria identified in this procedure.

- Form I: Sample Results Summary Form
- Form II: Initial and Continuing Calibration Verification Form
- Form III: Blanks Form
- Form V: Spike Sample Recovery Form
- Form VI: Duplicates Form
- Form VII: Laboratory Control Sample Form
- Form XIII: Preparation Log Form
- Form XIV: Analysis Run Log Form

Level C data validation consists of review of summary forms only while Level D data validation requires review of both summary forms and all associated raw data. Data review guidelines and how they apply to the different validation levels are indicated in the following text.

4.1 SAMPLE MANAGEMENT

QA/QC criteria included under sample management are sample preservation, handling, and transport; chain of custody (COC); and holding times.

4.1.1 Sample Preservation, Handling, and Transport

Level C and Level D:

Evaluate sample collection, handling, transport, and laboratory receipt from COC and laboratory receipt checklists to ensure that the samples have been properly handled. All samples must be stored at less than 6 degrees Celsius (°C). Water samples for the following analyses should be preserved as listed below or as specified in the analytical method:

- Chemical Oxygen Demand (COD) – Sulfuric acid to pH < 2
- Cyanide – NaOH to pH > 12
- Nitrate/Nitrite – Sulfuric acid to pH < 2
- Sulfide – Zinc acetate and NaOH to pH > 9
- Total Recoverable Petroleum Hydrocarbons – Sulfuric or hydrochloric acid to pH < 2
- Total Phosphorus – Sulfuric acid to pH < 2
- Total Organic Carbon (TOC) - Sulfuric or hydrochloric acid to pH < 2
- Total Organic Halides (TOX) – Sulfuric acid to pH < 2
- Total Hardness – Nitric acid to pH < 2

1. Any sample improperly preserved or arriving at the laboratory in a broken container shall be noted in the data validation report. If there is no indication of chemical preservation, assume samples are unpreserved. Professional judgment may result in the results of an analysis of an inappropriately preserved sample by the laboratory being qualified as estimated "J" or "UJ." In extreme cases (a preservation destructive to the analyte of interest) the sample data may be qualified as unusable, "R."
2. If any sample arriving at the laboratory for analysis is not refrigerated or the temperature of any cooler containing samples exceeds 4 ±2°C, this shall be noted in the data validation report; however, no qualification of data will be required.
3. If the temperature of the cooler was not recorded upon its receipt at the laboratory, document that the laboratory is noncompliant.

4.1.2 Chain of Custody

Level C and Level D:

Examine the COC for legibility and check that all wet chemistry analyses requested on the COC have been performed by the laboratory. Ensure that the COC Sample Number on the laboratory Form I matches the Sample Identification on the COC. Read the laboratory case narrative for additional information.

1. Verify collect dates, sampling times, and time zones. This is critical to evaluating parameters with short holding times.
2. Any samples received for analysis that were not analyzed shall be noted in the data validation report, along with the reason(s) for failure to analyze the samples, if the reason(s) can be determined. Conversely, samples that were analyzed for wet chemistry parameters but were not requested should also be noted.
3. Any discrepancies in sample naming between the COC and sample results form shall be noted in the data validation report with the correct sample name being identified if the correct sample name can be determined.
4. If the receiving laboratory transferred the samples to another laboratory for analysis, both the original COCs and transfer COCs shall be present. Document in the data validation report if the transfer COCs are not present.
5. Internal chain of custody is required for all samples, extracts, and digestates from receipt to disposal. Verify the internal COC forms for completeness. Document in the data validation report if the internal COC forms are not present.
6. Each individual cooler shall have an individual COC that lists only samples contained within that cooler. Document in the data validation report if multiple coolers appear on one COC.

4.1.3 Holding Times

Holding times for water samples shall be those given in the most recent version of 40 Code of Federal Regulations (CFR) Part 136 or SW-846, Volume 1, Section C, if not specified in 40 CFR, art 136, or those specified in the analytical method. Holding times are determined from the time of sample collection to the time of sample analysis. Water holding times will be applied to soil/sediment samples. Current water holding times are as follows:

- Alkalinity – 14 days
- Bromide – 28 days
- COD – 28 days
- Chloride – 28 days
- Chromium (VI) – 24 hours for unpreserved water samples (14 days from lab preservation); 28 days for soil samples
- Cyanide – 14 days
- Fluoride – 28 days
- Surfactants (M.B.A.S.) – 48 hours
- Nitrate – 48 hours for water samples; 28 days for soil samples
- Nitrite – 48 hours for water samples; 28 days for soil samples
- Nitrate/Nitrite – 28 days
- Orthophosphate – 48 hours for water samples; 28 days for soil samples
- Perchlorate – 28 days

- Sulfate – 28 days
- Sulfide – 7 days
- Total Phosphorus – 28 days
- Total Dissolved Solids – 7 days
- Total Suspended Solids – 7 days
- TOC – 28 days
- TOX – 28 days (7 days if not preserved)
- Total Recoverable Petroleum Hydrocarbons – 28 days
- pH – immediate upon sampling for water samples; 28 days for soil samples
- Total Hardness – 6 months

Level C and Level D:

If holding times are exceeded, flag all results greater than the detection limit (DL) or limit of quantitation (LOQ) as estimated "J" and all results less than the DL or LOQ as estimated "UJ" and document that holding times were exceeded. If holding times are grossly exceeded, the reviewer may determine that the data reported as nondetects are unusable "R." Data will not be qualified unusable "R" unless the holding time was exceeded by more than a factor of 2.

4.2 CALIBRATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run. Continuing calibration documents that the initial calibration is still valid and that maintenance and adjustment of the instrument on a day-to-day basis is satisfactory.

4.2.1 The Initial Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run.

Level C and Level D:

A blank and at least three standards must be used in establishing the analytical curve.

If the correlation coefficient is below 0.995, qualify all associated detects as estimated "J" and all nondetects as "UJ." If the correlation coefficient is significantly lower than 0.995, professional judgment may be used to reject, "R," the analytes associated with the initial calibration.

Level D:

Recalculate the correlation coefficient for all initial calibrations. Verify from the raw data that appropriate concentration and number of standards were utilized to establish analytical curves and the associated correlation coefficients.

4.2.2 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

The working calibration curve must be verified at the interval of 10 percent to ensure that the system performance has not degraded. Continuing calibration documents that the initial calibration is still valid and that maintenance and adjustment of the instrument on a day-to-day basis is satisfactory. Calibration must be verified with an independently prepared check standard.

Level C and Level D:

Review the ICV and CCV percent recovery (%R) forms. Analysis results must fall within the control limits of 90–110 percent recovery of the true value except perchlorate. Analysis results for perchlorate must fall within the control limits of 75–125 percent recovery of the true value for the ICV and 85–115 percent recovery of the true value for the CCV.

1. Due to possible rounding discrepancies, allow the results to fall within 1 percent of the acceptance windows (e.g., 89–111 percent).
2. If after a failing CCV, two additional consecutive CCVs are analyzed immediately, and both additional CCVs are within the control limits, the data is acceptable. If either of the additional CCVs is not within control limits, then the associated data will need qualification. See below for the recommended qualification guidelines.
3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the technical review. The following guidelines are recommended:
   a. If the ICV or CCV %R falls outside the acceptance windows but within the ranges of 75–89 percent or 111–125 percent (perchlorate, 70–84 percent or 116–130 percent), qualify results greater than the DL as estimated "J."
   b. If the ICV or CCV %R is within the range of 111–125 percent (perchlorate, 116-130 percent), results less than the DL are acceptable.
   c. If the ICV or CCV %R is 75–89 percent (perchlorate, 70–84 percent), qualify results less than the DL as nondetected and estimated "UJ."
   d. If the ICV or CCV %R is less than 75 percent (perchlorate, less than 70 percent), qualify all results as unusable "R."
   e. If the ICV or CCV %R is greater than 125 percent (perchlorate, greater than 130 percent), qualify results greater than the DL as unusable "R"; results less than the DL are acceptable.

Level D:

1. Recalculate and verify one or more of the ICV and CCV %Rs per type of analysis using the following equation for %R. Once again, to correct for possible rounding discrepancies, let the results fall within 1 percent of the contract windows (e.g., 89–111 percent).

$$\%\text{Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Concentration (in micrograms per liter [µg/L]) of each analyte measured in the analysis of the ICV or CCV solution.

Q_a = Concentration (in µg/L) of each analyte in the ICV or CCV source.

2. If discrepancies are discovered on any form, request a resubmittal from the laboratory and validate according to the criteria outlined above.

4.3 BLANKS

Method (preparation) and calibration blank analyses results are assessed to determine the existence and magnitude of laboratory contamination problems. If problems with any blank exist, all data associated with the blank must be carefully evaluated to determine whether there is a bias on the data, or if the problem is an isolated occurrence not affecting data.

4.3.1 Calibration Blanks

Level C and Level D:

1. If the blank is less than the LOQ and the samples results are greater than the DL but less than the LOQ, then qualify "U" at the result.
2. If the blank is less than the LOQ and the sample results are greater than the LOQ or nondetect, the data is acceptable.
3. If the blank is greater than the LOQ, then samples less than 5x the blank will be qualified as "U" at the concentration. Samples greater than 5x the blank are acceptable.
4. For negative blanks where the absolute value of the blank is greater than the LOQ, sample results that are less than 10x the absolute value of the negative blank qualify "J" for detect and 'UJ" for nondetect results. Results that are greater than 10x the absolute value of the negative blank are acceptable.

Ensure that units are correct when applying calibration blank qualifications. If samples are soil matrix, results must first be converted to µg/L from milligrams per kilogram to make correct comparisons.

Level D:

Verify one or more of the calibration blank results per type of analysis by comparing the Form III to the raw data. After the validity of the target analytes are verified, validate the corresponding data using the criteria outlined above for Level C and Level D validation.

4.3.2 Method (Preparation) Blanks

Level C and Level D:

At least one method blank must be prepared with each batch of samples. If a method blank was not prepared and analyzed as required, the reviewer may qualify associated sample results less than the DL as nondetected and estimated "UJ," and sample results greater than the MDL as estimated "J."

Professional judgment should be utilized, however, taking into account the results of other associated blanks (e.g., initial calibration blank, continuing calibration blank).

If analytes of interest are detected in the method blanks, the procedure for the qualification of associated sample results is identical to the rules outlined in Section 4.3.1 of this procedure.

Level D:

Verify out-of-control method blanks that result in the qualification of numerous analytes against the raw data. Verify the results reported on Form III. After the validity of the target analytes are verified, validate the corresponding data using the criteria outlined above for Level C and Level D validation.

4.4 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

Blank spike/laboratory control sample (LCS) recoveries must be within the QSM Appendix C limits specified in the DoD QSM unless project-specific control limits are established for a given sample matrix. Use in-house limits if analytes are not listed in Appendix C or project limits are not specified.

Level C and Level D:

1. If the blank spike/LCS results are less than 50 percent, only the spiked analytes that showed low recovery in all associated samples shall be flagged as "R" for nondetects and "J" for detects.
2. If blank spike/LCS results are below the control limits (but above 50 percent), spiked analytes that showed low recovery in all associated samples shall be flagged as estimated "UJ" or "J."
3. If blank spike/LCS results are above the control limits, detects for only the spiked analytes that showed high recovery in all associated samples shall be flagged as "J."
4. If the laboratory analyzes a blank spike duplicate/LCS duplicate (LCSD), evaluate and qualify the LCSD results using the criteria noted above.
5. If the relative percent differences (RPDs) between LCS and LCSD results are above the control limits (use the matrix spike [MS]/matrix spike duplicate [MSD] RPD control limits identified in DoD QSM Appendix B, if none are available use laboratory in-house limits), spiked compounds which showed high RPD in all associated samples shall be flagged as estimated "UJ" or "J."

Level D:

To check that the spike %R was calculated and reported correctly using the following equation, recalculate one spike recovery per matrix (and any spike that would result in the qualification of a sample).

$$\%\text{Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Quantity determined by analysis

Q_a = Quantity added to samples/blanks

If transcription errors are discovered on Form VII (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.5 MATRIX SPIKE/MATRIX SPIKE DUPLICATE AND MATRIX DUPLICATE

MS/MSD and matrix duplicate (MD) data are used to determine the effect of the matrix on a method's recovery efficiency and precision for a specific sample matrix. MD analyses are also performed to demonstrate acceptable method precision by the laboratory at the time of analysis.

If the MS/MSD and MD results do not meet the technical criteria, apply the action to the source sample only.

MS/MSD results should be within the QC limits specified in the DoD QSM Appendix C unless project-specific control limits are established for a given sample matrix. Use in-house limits if spiked analytes are not listed in Appendix C or project limits are not specified.

For the MD RPD, samples greater than 5× the LOQ use RPD to evaluate. For samples less than 5× the LOQ, use the difference between the MD and the sample unless project limits are specified. For difference use 1× the LOQ as the control limit for water samples and 2× the LOQ as the control limit for soil samples unless project limits are specified.

Level C and Level D:

1. The laboratory must spike and analyze a MS/MSD or MD from the specific project site as required for each matrix type and analytical batch.
2. MS/MSD data should be reported on a MS/MSD summary form similar to Form V. MD data should be reported on a MD summary form similar to Form VI.
3. If MS/MSD results are below the control limits, spiked analytes that showed low recovery shall be flagged as estimated "UJ" or "J."
4. If MS/MSD results are above the control limits, detects for only the spiked analytes that showed high recovery shall be flagged as "J."
5. If the RPD between MS and MSD recoveries or the RPD or difference between the MD and sample are greater than 15 percent, qualify the sample as estimated "UJ" or "J."
6. Failure of MS/MSD due to the presence of a target analyte in the parent sample at greater than four times the spike concentration should not result in any qualifications. Note the incident in the data validation report.

Level D:

Check the raw data and recalculate one or more %Rs, especially %Rs that resulted in the qualification of data, using the following equation to verify that results on Forms V and VI (or equivalent) are correct.

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

$$RPD = \frac{ABS|SSR - SDR|}{(SSR + SDR)/2} \times 100$$

Where:

SA	=	spike added
SR	=	sample result
SSR	=	spiked sample result
SDR	=	spiked duplicate result
ABS	=	absolute value

If transcription errors are discovered on Forms V or VI (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.6 SAMPLE RESULT VERIFICATION

Level C:

Level C validation does not require the evaluation of raw data, sample result verification is not required. All soil sample results are reported on a dry weight basis.

Level D:

The raw data should be examined to verify that the correct calculation of the sample results was reported by the laboratory. Sample preparation logs, instrument printouts, strip charts, etc. should be compared to the reported sample results recorded on the sample results summary forms. All soil sample results are reported on a dry weight basis.

1. Evaluate the raw data for any anomalies (i.e., baseline shifts, negative absorbance, omissions, legibility).
2. Verify that there are no errors in transcription or calculation. If errors are discovered, request a resubmittal from the laboratory and validate the data according the criteria outlined above.
3. Verify that results fall within the calibrated range. If the positive sample result falls outside the calibrated range, qualify the sample result "J."

4.7 FIELD QC SAMPLES

Field QC samples discussed in this section of the procedure are equipment blanks, field blanks, field duplicates, and field triplicates. Analytical results for field QC samples are utilized to qualify associated sample results.

4.7.1 Equipment Blanks and Field Blanks

Analytes detected in equipment blanks indicate the possibility of cross-contamination between samples due to improper equipment decontamination.

A field blank sample may be collected from each source of water used during each sampling event. The field blank may be analyzed to assess whether the chemical nature of the water used in decontamination may have affected the analytical results of site samples.

If analytes are detected in the equipment blanks and/or field blanks, the procedure for the qualification of associated sample results is identical to the criteria outlined in Section 4.3.1 of this procedure.

Level C and Level D:

1. Determine which field QC samples apply to samples in the sample delivery group.
2. Ensure that units are correct when applying field QC blank qualifications. If samples are soil matrix, results must first be converted to µg/L from micrograms per kilogram to make correct comparisons.
3. Because of the way in which the field blanks and equipment blanks are sampled, equipment blanks are not qualified because of field blank contamination. The affected samples are qualified, however, by either the field blank or equipment blank results, whichever has the higher contaminant concentration.
4. Equipment blanks and field blanks are only qualified with method blank results in order to account for laboratory contamination.

Level D:

1. Verify all target analytes found in the equipment blanks and field blanks against the raw data.
2. After the validity of the target analytes are verified, validate the corresponding data using the criteria outlined above for Level C and Level D validation.

4.7.2 Field Duplicates and Field Triplicates

Field duplicates consist of either collocated or subsampled samples. Field duplicates for ground water and surface water samples are generally considered to be collocates. Soil duplicate samples may be homogenized and subsampled in the field (or at the laboratory) to form an original and duplicate sample, or may be an additional volume of sample collected in a separate sample container to form a collocate sample. Field duplicate results are an indication of both field and laboratory precision; the results may be used to evaluate the consistency of sampling practices.

Field triplicates are collected from different, randomly selected locations to verify that an incremental sample truly represents a decision unit. Field triplicate results are more useful than field duplicates to statistically evaluate sampling precision.

Level C and Level D:

1. Check to ensure that field duplicates and/or field triplicates were collected and analyzed as specified in the project planning documents. If the sampling frequency is less than the frequency stated in the planning documents, no qualification of the associated sample results is necessary but the incident shall be discussed in the data validation report.

2. For field duplicate results, if the RPDs are greater than 50 percent for water or 100 percent for soil or as stated in the planning document if more conservative, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.
3. For field triplicate results, if the RSDs are greater than the QC limits stated in the planning document, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.

Level D:

Verify by recalculating at least two detects common between the sample and its field duplicate and/or field triplicate. If discrepancies are discovered, document in the data validation report.

5. Records

A Form I that has been validated and verified, and has been determined by the data validator to accurately represent the appropriate sample results to be utilized, shall be stamped "NAVFAC PACIFIC VALIDATED." Additionally, sample result forms for which the data has been validated at the Level D validation level shall be stamped or noted "Level D."

Copies of all documents generated by the data validation personnel will be stored for no less than 10 years.

6. References

American Public Health Association (APHA). 2005. *Standard Methods for the Examination of Water and Wastewater.* 21st ed. Washington. September.

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities.* Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www2.epa.gov/sites/production/files/documents/qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets.* Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Environmental Protection Agency, United States (EPA). 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Final Update IV. Office of Solid Waste. Updates available: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

Procedure II-A, *Data Validation*.

7. Attachments

None.

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Data Quality Assessment Report

1. Purpose

This procedure describes the presentation format and information provided in the data quality assessment report (DQAR) under the United States (U.S.) Navy Environmental Restoration (ER) Program for Naval Facilities Engineering Command (NAVFAC), Pacific and is consistent with protocol in the *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories* (DoD QSM) (DoD 2013). The objective of DQAR is to summarize the validated data to the end user. This procedure also establishes the method by which a Contract task Order (CTO) Manager selects and confirms the content of the DQAR. Data validation is addressed separately in Procedure II-A, *Data Validation.*

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Responsibilities

The CTO Manager, the QA Manager or Technical Director, and the CTO QA Coordinator are responsible for ensuring that this procedure is implemented by data validation personnel.

Data validation personnel (unless otherwise stated) are responsible for implementing this procedure for all DQARs.

4. Procedure

4.1 Introduction

The DQAR summarizes the QA/quality control (QC) evaluation of the data according to precision, accuracy, representativeness, completeness, comparability, and sensitivity relative to the project quality objectives (PQOs). The report provides a quantitative and qualitative assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall usability.

The DQAR summary report identifies the level of data validation for each sample and evaluates and summarizes the results of QA/QC data validation for the entire sampling program. Each analytical fraction has a separate section for each of the criteria. These sections interpret specific QC deviations and their effects on both individual data points and the analyses as a whole. The last section presents

a summary of the precision, accuracy, representativeness, completeness, comparability, and sensitivity criteria by comparing quantitative parameters with acceptability criteria defined in the PQOs. Qualitative criteria are also summarized in this section. A DQAR example is provided as Attachment II-S-1.

4.2 PRECISION AND ACCURACY OF ENVIRONMENTAL DATA

Environmental data quality depends on sample collection procedures, analytical methods and instrumentation, documentation, and sample matrix properties. Both sampling procedures and laboratory analyses contain potential sources of uncertainty, error, and/or bias, which affect the overall quality of a measurement. Errors in sample data may result from incomplete equipment decontamination, inappropriate sampling techniques, sample heterogeneity, improper filtering, and improper preservation. The accuracy of analytical results is dependent on selecting appropriate analytical methods, maintaining equipment properly, and complying with QC requirements. The sample matrix also is an important factor in the ability to obtain precise and accurate results within a given media.

Environmental and laboratory QC samples assess the effects of sampling procedures and evaluate laboratory contamination, laboratory performance, and matrix effects. QC samples include: trip blanks, equipment blanks, field blanks, field duplicates, field triplicates, method blanks, laboratory control samples (LCSs), surrogate spikes, matrix spike/matrix spike duplicates (MS/MSDs), laboratory duplicates, and laboratory triplicates.

Before producing the DQAR, the analytical data should be validated according to the NAVFAC Pacific data validation procedures. Samples not meeting the NAVFAC ER Program validation criteria are qualified with a flag, an abbreviation indicating a deficiency with the data. The following are flags used in data validation.

J Estimated. The associated numerical value is an estimated quantity. The analyte was detected but the reported value may not be accurate or precise. The "J" qualification indicates the data fell outside the QC limits, but the exceedance was not sufficient to cause rejection of the data.

R Rejected. The data is unusable (the compound or analyte may or may not be present). Use of the "R" qualifier indicates a significant variance from functional guideline acceptance criteria. Either resampling or re-analysis is necessary to determine the presence or absence of the rejected analyte.

U Nondetected. Analyses were performed for the compound or analyte, but it was not detected. The "U" designation is also applied to suspected blank contamination. The "U" flag is used to qualify any result detected in an environmental sample at a concentration less than 10 times the value of the concentration in any associated blank for common laboratory contaminants and less than 5 times the concentration in any associated blank for all other contaminants.

UJ Estimated/Nondetected. Analyses were performed for the compound or analyte, but it was not detected and the limit of detection (LOD) is an estimated quantity due to poor accuracy or precision. This qualification is also used to flag possible false negative results in the case where low bias in the analytical system is indicated by low calibration response, surrogate, internal standard, or other spike recovery.

Once the data are reviewed and qualified according to the NAVFAC Pacific data validation procedures, the data set is then evaluated using precision, accuracy, representativeness, completeness, comparability, and sensitivity criteria that provide an evaluation of overall data usability. The following is a discussion of the precision, accuracy, representativeness, completeness, and comparability criteria as related to the PQOs.

4.2.1 Precision

Precision is a measure of the agreement or reproducibility of analytical results under a given set of conditions. It is a quantity that cannot be measured directly but is calculated from reported concentrations. Precision is expressed as the relative percent difference (RPD) or percent relative standard deviation (%RSD):

$$RPD = (D1-D2)/\{1/2(D1+D2)\} \times 100$$

$$\%RSD = SD/\{1/3(D1+D2+D3)\} \times 100$$

Where:

D1	=	the reported concentration for primary sample analyses
D2	=	the reported concentrations for duplicate analyses
D3	=	the reported concentrations for triplicate analyses
SD	=	the standard deviation for sample, duplicate and triplicate analyses

Precision is primarily assessed by calculating a RPD from the reported concentrations of the spiked compounds for each sample in the MS/MSD pair. In the absence of a MS/MSD pair, a laboratory duplicate or LCS/laboratory control sample duplicate (LCSD) pair can be analyzed as an alternative means of assessing precision. In some cases, samples from multiple sample delivery groups (SDGs) are within one QC batch and therefore are associated with the same laboratory QC samples. An additional measure of sampling precision may be obtained by collecting and analyzing field duplicate samples, which are compared using the RPD result as the evaluation criteria.

MS and MSD samples are field samples spiked by the laboratory with target analytes prior to preparation and analysis. These samples measure the overall efficiency of the analytical method in recovering target analytes from an environmental matrix. A LCS is similar to a MS/MSD sample in that the LCS is spiked with the same target analytes prior to preparation and analysis. However, the LCS is prepared using a controlled interference-free matrix instead of a field sample aliquot. Laboratory reagent water is used to prepare aqueous LCS. Non-aqueous LCSs are prepared using solid media approved by the American Society for Testing and Materials for their homogeneity. The LCS measures laboratory efficiency in recovering target analytes from either a solid or aqueous matrix in the absence of matrix interferences.

For inorganic analysis, one primary sample is analyzed and accompanied by an unspiked laboratory duplicate. The data reviewer compares the reported results of the primary analysis and the laboratory duplicate and calculates RPDs to assess laboratory precision.

Laboratory and field sampling precision are further evaluated by calculating RPDs for field sample duplicate pairs. The sampler collects two field samples at the same location and under identically controlled conditions. The laboratory then analyzes the samples under identical conditions.

If incremental sampling is performed, laboratory and field sampling precision are evaluated by calculating RSDs for laboratory triplicates and field triplicates. At the subsampling step, one sample is prepared in triplicate per batch. Laboratory triplicate data are used to determine that the samples are being reduced to sufficiently small particle sizes during the grinding process. Field triplicates are collected from different, randomly selected locations to verify that an incremental sample truly represents a decision unit. Field triplicate results are more useful than field duplicates to statistically evaluate sampling precision.

An RPD outside the numerical QC limit in either MS/MSD samples or LCS/LCSD or a %RSD outside the numerical QC limit in the laboratory triplicate indicates imprecision. Imprecision is the variance in the consistency with which the laboratory arrives at a particular reported result. Thus, the actual analyte concentration may be higher or lower than the reported result.

Possible causes of poor precision include sample matrix interference, improper sample collection or handling, inconsistent sample preparation, and poor instrument stability. In some duplicates and/or triplicates, results may be reported in the primary, duplicate, or triplicate samples at levels below the limit of quantitation (LOQ) or non-detected. Since these values are considered to be estimates, RPD exceedances from duplicates or %RSD exceedances from triplicates do not suggest a significant impact on the data quality.

4.2.2 Accuracy

Accuracy is a measure of the agreement of an experimental determination and the true value of the parameter being measured. It is used to identify bias in a given measurement system. Recoveries outside acceptable QC limits may be caused by factors such as instrumentation, analyst error, or matrix interference. Accuracy is assessed through the analysis of MS, MSD, LCS, and samples containing surrogate spikes. In some cases, samples from multiple SDGs are within one QC batch and therefore are associated with the same laboratory QC samples. Surrogate spikes are either isotopically labeled compounds or compounds that are not typically detected in the samples. Surrogate spikes are added to every blank, environmental sample, MS/MSD, and standard, for applicable organic analyses. Accuracy of inorganic analyses is determined using the percent recoveries of MS and LCS analyses.

Percent recovery (%R) is calculated using the following equation:

$$\%R = (A-B)/C \times 100$$

Where:

A	=	measured concentration in the spiked sample
B	=	measured concentration of the spike compound in the unspiked sample
C	=	concentration of the spike

The percent recovery of each analyte spiked in MS/MSD samples, LCS, and surrogate compounds added to environmental samples is evaluated against the acceptance criteria specified by the previously noted documents. Spike recoveries outside the acceptable QC accuracy limits provide an indication of bias, where the reported data may overestimate or underestimate the actual concentration of compounds detected or quantitation limits reported for environmental samples.

4.3 REPRESENTATIVENESS

Representativeness is a qualitative parameter that expresses the degree to which the sample data are characteristic of a population and is evaluated by reviewing the QC results of blank samples and holding times. Positive detects of compounds in the blank samples identify compounds that may have been introduced into the samples during sample collection, transport, preparation, or analysis. The various types of blanks evaluated are discussed below.

A method blank is a laboratory grade water or solid matrix that contains the method reagents and has undergone the same preparation and analysis as the environmental samples. The method blank provides a measure of the combined contamination derived from the laboratory source water, glassware, instruments, reagents, and sample preparation steps. Method blanks are prepared for each sample of a similar matrix extracted by the same method at a similar concentration level.

For inorganic analyses, initial and continuing calibration blanks consist of acidified laboratory grade water, which are injected at the beginning and at a regular frequency during each 12-hour sample analysis run. These blanks estimate residual contaminants from the previous sample or standards analysis and measure baseline shifts that commonly occur in emission and absorption spectroscopy.

Trip blanks are used to identify possible volatile organic contamination introduced into the sample during transport. A trip blank is a sample volatile organics analysis vial filled in the laboratory with reagent-grade water and preserved to a pH less than 2 with hydrochloric acid. It is transported to the site, stored with the sample containers, and returned unopened to the laboratory for analysis.

Equipment blanks consist of analyte-free water poured over or through the sample collection equipment. The water is collected in a sample container for laboratory analysis. These blanks are collected after the sampling equipment is decontaminated and measure efficiency of the decontamination procedure.

Field blanks consist of analyte-free source water stored at the sample collection site. The water is collected from each source water used during each sampling event.

If sample grinding is performed, grinding blanks, which consist of clean solid matrix (such as Ottawa sand), must be prepared (e.g., ground and subsampled) and analyzed in the same manner as a field sample. Grinding equipment must be thoroughly cleaned between the processing of samples and grinding blanks must be processed and analyzed to prevent cross-contamination.

Contaminants found in both the environmental sample and a blank sample are assumed to be laboratory artifacts if the concentration in the environmental sample is less than 10 times the blank value for common laboratory contaminants (methylene chloride, acetone, 2-butanone, and phthalate esters) or 5 times the blank value for other laboratory contaminants.

Holding times are evaluated to assure that the sample integrity is intact for accurate sample preparation and analysis. Holding times will be specific for each method and matrix analyzed. Holding time exceedances can cause loss of sample constituents due to biodegradation, precipitation, volatization, and chemical degradation.

4.4 COMPARABILITY

Comparability is a qualitative expression of the confidence with which one data set may be compared to another. It provides an assessment of the equivalence of the analytical results to data obtained from other analyses. It is important that data sets be comparable if they are used in conjunction with other data sets. The factors affecting comparability include the following: sample collection and handling techniques, matrix type, and analytical method. If these aspects of sampling and analysis are carried out according to standard analytical procedures, the data are considered comparable. Comparability can only be compared with confidence when precision, accuracy, and representativeness are known.

4.5 COMPLETENESS

Completeness is defined as the percentage of acceptable sample results compared to the total number of sample results. Completeness is evaluated to determine if an acceptable amount of usable data were obtained so that a valid scientific site assessment can be completed. Completeness equals the total number of sample results for each fraction minus the total number of rejected sample results divided by the total number of sample results multiplied by 100. The goal for completeness for target analytes in each analytical fraction should be specified in the DoD QSM (DoD 2013) or project planning document.

Percent completeness is calculated using the following equation:

$$\%C = (T - R)/T \times 100$$

Where:

%C	=	percent completeness
T	=	total number of sample results
R	=	total number of rejected sample results

Completeness is also determined by comparing the planned number of samples per method and matrix as specified in the project planning document, with the number determined above.

4.6 SENSITIVITY

Sensitivity is the ability of an analytical method or instrument to discriminate between measurement responses representing different concentrations. This capability is established during the planning phase to meet the data quality objectives (DQOs). It is important that calibration requirements, detection limits (DLs), and project-specific LODs and LOQs presented in the work plan are achieved and that target analytes can be detected at concentrations necessary to support the DQOs. In addition, sample results are compared to method blank and field blank results to identify potential effects of laboratory background and field procedures on sensitivity.

5. References

Department of Defense, United States (DoD). 2013. *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories.* Final version 5.0. Prepared by Department of Defense Environmental Data Quality Workgroup and the Department of Energy Consolidated Audit Program Operations Team. March.

Procedure II-A, *Data Validation*.

6. Attachments

II-S-1: Data Quality Assessment Report Example

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Attachment II-S-1
Data Quality Assessment Report Example

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DATA QUALITY ASSESSMENT REPORT

SITE INVESTIGATION BUILDING E-13
PEARL HARBOR, CTO XXX

12/1/03

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Glossary

µg/kg	microgram per kilogram
µg/L	microgram per liter
BTEX	benzene, toluene, ethylbenzene, xylenes
DL	detection limit
DQO	data quality objectives
EPA	Environmental Protection Agency, United States
IDL	instrument detection limit
LCS/LCSD	laboratory control sample/laboratory control sample duplicate
LOD	limit of detection
LOQ	limit of quantitation
mg/kg	milligram per kilogram
MS/MSD	matrix spike/matrix spike duplicate
NAS	Naval Air Station
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness, Sensitivity
PAH	polynuclear aromatic hydrocarbon
PCB	polychlorinated biphenyl
QA/QC	quality assurance/quality control
RPD	relative percent difference
RRF	relative response factor
RL	reporting limit
SDG	sample delivery group
%D	percent difference
%R	percent recovery
%RSD	percent relative standard deviation

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1. Introduction

A remediation and closure was conducted at Building E-13 at Pearl Harbor, Oahu, Hawaii. This part of the site investigation included the collection and analyses of 141 environmental and quality control (QC) samples. The analyses were performed by the following methods:

- Polynuclear aromatic hydrocarbons (PAHs) by United States Environmental Protection Agency (EPA) SW-846 8270C-SIM
- Polychlorinated biphenyls (PCBs) by EPA SW-846 Method 8082
- Metals by EPA SW-846 Method 6010B/6020/7471A

Analytical services were provided by ZZZZ Laboratories whom performed analyses on the water and soil samples. The samples were grouped into sample delivery groups (SDGs) of up to 20 field samples received by each laboratory. The environmental samples are associated with QA/QC samples designed to document the data quality of the entire SDG or a sub-group of samples within a SDG. Table I is a cross-reference table listing each sample, analysis, SDG, collection date, laboratory sample number, and matrix. All shaded samples in Table I were reviewed under Level D validation guidelines.

One hundred percent of the analytical data were validated according to NAVFAC Pacific Level D data validation procedures. The analytical data were evaluated for quality assurance and quality control (QA/QC) based on the *Department of the Navy Environmental Restoration Program (NERP) Manual* (2006).

This data quality assessment report (DQAR) summarizes the QA/QC evaluation of the data according to precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) relative to the project quality objectives (PQOs). This report provides a quantitative and qualitative assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall usability.

The DQAR evaluates and summarizes the results of QA/QC data validation for the entire sampling program. Each analytical fraction has a separate section for each of the PARCC criteria. These sections interpret specific QC deviations and their effects on both individual data points and the analyses as a whole. Section 6 presents a summary of the PARCC criteria by comparing quantitative parameters with acceptability criteria defined in the PQOs. Qualitative PARCC criteria are also summarized in this section.

Precision and Accuracy of Environmental Data

Environmental data quality depends on sample collection procedures, analytical methods and instrumentation, documentation, and sample matrix properties. Both sampling procedures and laboratory analyses contain potential sources of uncertainty, error, and/or bias, which affect the overall quality of a measurement. Errors in sample data may result from incomplete equipment decontamination, inappropriate sampling techniques, sample heterogeneity, improper filtering, and improper preservation. The accuracy of analytical results is dependent on selecting appropriate analytical methods, maintaining equipment properly, and complying with QC requirements. The sample matrix also is an important factor in the ability to obtain precise and accurate results within a given media.

Environmental and laboratory QA/QC samples assess the effects of sampling procedures and evaluate laboratory contamination, laboratory performance, and matrix effects. QA/QC samples include: equipment blanks, field duplicates, method blanks, laboratory control samples (LCSs), surrogate spikes, matrix spike/matrix spike duplicates (MS/MSDs), and laboratory duplicates.

Before conducting the PARCC evaluation, the analytical data were validated according to the *Department of the Navy Environmental Restoration Program (NERP) Manual* [2006]). Samples not meeting the Project Procedures Manual acceptance criteria were qualified with a flag, an abbreviation indicating a deficiency with the data. The following are flags used in data validation.

J — Estimated: The associated numerical value is an estimated quantity. The analyte was detected but the reported value may not be accurate or precise. The "J" qualification indicates the data fell outside the QC limits, but the exceedance was not sufficient to cause rejection of the data.

R — Rejected: The data is unusable (the compound or analyte may or may not be present). Use of the "R" qualifier indicates a significant variance from functional guideline acceptance criteria. Either resampling or re-analysis is necessary to determine the presence or absence of the rejected analyte.

U — Nondetected: Analyses were performed for the compound or analyte, but it was not detected. The "U" designation is also applied to suspected blank contamination. The "U" flag is used to qualify any result detected in an environmental sample at a concentration less than 10 times the value of the concentration in any associated blank for common laboratory contaminants and less than 5 times the concentration in any associated blank for all other contaminants.

UJ — Estimated/Nondetected: Analyses were performed for the compound or analyte, but it was not detected and the limit of detection (LOD) is an estimated quantity due to poor accuracy or precision. This qualification is also used to flag possible false negative results in the case where low bias in the analytical system is indicated by low calibration response, surrogate, internal standard, or other spike recovery.

Once the data are reviewed and qualified according to the *Department of the Navy Environmental Restoration Program (NERP) Manual* (2006), the data set is then evaluated using PARCCS criteria. PARCCS criteria provide an evaluation of overall data usability. The following is a discussion of PARCCS criteria as related to the PQOs.

Precision is a measure of the agreement or reproducibility of analytical results under a given set of conditions. It is a quantity that cannot be measured directly but is calculated from reported concentrations. Precision is expressed as the relative percent difference (RPD):

$$RPD = (D1-D2)/\{1/2(D1+D2)\} \times 100$$

Where:

D1 and D2 = the reported concentrations for sample and duplicate analyses.

Precision is primarily assessed by calculating a RPD from the reported concentrations of the spiked compounds for each sample in the MS/MSD pair. In the absence of a MS/MSD pair, a laboratory

duplicate or LCS/LCSD pair can be analyzed as an alternative means of assessing precision. In some cases, samples from multiple SDGs were within one QC batch and therefore are associated with the same laboratory QC samples. An additional measure of sampling precision was obtained by collecting and analyzing field duplicate samples, which were compared using the RPD result as the evaluation criteria.

MS and MSD samples are field samples spiked by the laboratory with target analytes prior to preparation and analysis. These samples measure the overall efficiency of the analytical method in recovering target analytes from an environmental matrix. A LCS is similar to a MS/MSD sample in that the LCS is spiked with the same target analytes prior to preparation and analysis. However, the LCS is prepared using a controlled interference-free matrix instead of a field sample aliquot. Laboratory reagent water is used to prepare aqueous LCS. Non-aqueous LCSs are prepared using solid media approved by the American Society for Testing and Materials (ASTM) for their homogeneity. The LCS measures laboratory efficiency in recovering target analytes from either a solid or aqueous matrix in the absence of matrix interferences.

For inorganics analysis, one primary sample is analyzed and accompanied by an unspiked laboratory duplicate. The data reviewer compares the reported results of the primary analysis and the laboratory duplicate, then calculates RPDs, which are used to assess laboratory precision.

Laboratory and field sampling precision are further evaluated by calculating RPDs for aqueous field sample duplicate pairs. The sampler collects two field samples at the same location and under identically controlled conditions. The laboratory then analyzes the samples under identical conditions.

An RPD outside the numerical QC limit in either MS/MSD samples or LCS/LCSD indicates imprecision. Imprecision is the variance in the consistency with which the laboratory arrives at a particular reported result. Thus, the actual analyte concentration may be higher or lower than the reported result.

Possible causes of poor precision include sample matrix interference, improper sample collection or handling, inconsistent sample preparation, and poor instrument stability. In some duplicate pairs, results maybe reported in either the primary or duplicate samples at levels below the limit of quantitation (LOQ) or non-detected. Since these values are considered to be estimates, RPD exceedances from these duplicate pairs do not suggest a significant impact on the data quality.

Accuracy is a measure of the agreement of an experimental determination and the true value of the parameter being measured. It is used to identify bias in a given measurement system. Recoveries outside acceptable QC limits may be caused by factors such as instrumentation, analyst error, or matrix interference. Accuracy is assessed through the analysis of MS, MSD, LCS, and samples containing surrogate spikes. In some cases, samples from multiple SDGs were within one QC batch and therefore are associated with the same laboratory QC samples. Surrogate spikes are either isotopically labeled compounds or compounds that are not typically detected in the samples. Surrogate spikes are added to every blank, environmental sample, MS/MSD, and standard, for all applicable organic analyses. Accuracy of inorganic analyses is determined using the percent recoveries of MS and LCS analyses.

%R is calculated using the following equation:

$$\%R = (A-B)/C \times 100$$

Where:

A	=	measured concentration in the spiked sample
B	=	measured concentration of the spike compound in the unspiked sample
C	=	concentration of the spike

The percent recovery of each analyte spiked in MS/MSD samples, LCS, and surrogate compounds added to environmental samples is evaluated against the acceptance criteria specified by the previously noted documents. Spike recoveries outside the acceptable QC accuracy limits provide an indication of bias, where the reported data may overestimate or underestimate the actual concentration of compounds detected or quantitation limits reported for environmental samples.

Representativeness is a qualitative parameter that expresses the degree to which the sample data are characteristic of a population and is evaluated by reviewing the QC results of blank samples and holding times. Positive detects of compounds in the blank samples identify compounds that may have been introduced into the samples during sample collection, transport, preparation, or analysis. The QA/QC blanks collected and analyzed are method blanks.

A method blank is a laboratory grade water or solid matrix that contains the method reagents and has undergone the same preparation and analysis as the environmental samples. The method blank provides a measure of the combined contamination derived from the laboratory source water, glassware, instruments, reagents, and sample preparation steps. Method blanks are prepared for each sample of a similar matrix extracted by the same method at a similar concentration level.

For inorganic analyses, initial and continuing calibration blanks consist of acidified laboratory grade water, which are injected at the beginning and at a regular frequency during each 12 - hour sample analysis run. These blanks estimate residual contaminants from the previous sample or standards analysis and measure baseline shifts that commonly occur in emission and absorption spectroscopy.

Trip blanks are used to identify possible volatile organic contamination introduced into the sample during transport. A trip blank is a sample bottle filled in the laboratory with reagent-grade water and preserved to a pH less than 2 with hydrochloric acid. It is transported to the site, stored with the sample containers, and returned unopened to the laboratory for analysis.

Equipment blanks consist of analyte-free water poured over or through the sample collection equipment. The water is collected in a sample container for laboratory analysis. These blanks are collected after the sampling equipment is decontaminated and measure efficiency of the decontamination procedure. Equipment blanks were collected and analyzed for all target analytes.

Field blanks consist of analyte-free source water stored at the sample collection site. The water is collected from each source water used during each sampling event. Field blanks were collected and analyzed for all target analytes.

Contaminants found in both the environmental sample and a blank sample are assumed to be laboratory artifacts if the concentration in the environmental sample is less than 10 times the blank

value for common laboratory contaminants; methylene chloride, acetone, 2-butanone, and phthalate esters or 5 times the blank value for other laboratory contaminants.

Holding times are evaluated to assure that the sample integrity is intact for accurate sample preparation and analysis. Holding times will be specific for each method and matrix analyzed. Holding time exceedances can cause loss of sample constituents due to biodegradation, precipitation, volatization, and chemical degradation.

Comparability is a qualitative expression of the confidence with which one data set may be compared to another. It provides an assessment of the equivalence of the analytical results to data obtained from other analyses. It is important that data sets be comparable if they are used in conjunction with other data sets. The factors affecting comparability include the following: sample collection and handling techniques, matrix type, and analytical method. If these aspects of sampling and analysis are carried out according to standard analytical procedures, the data are considered comparable. Comparability is also dependent upon other PARCC criteria, because only when precision, accuracy, and representativeness are known can data sets be compared with confidence.

Completeness is defined as the percentage of acceptable sample results compared to the total number of sample results. Completeness is evaluated to determine if an acceptable amount of usable data were obtained so that a valid scientific site assessment can be completed. Completeness equals the total number of sample results for each fraction minus the total number of rejected sample results divided by the total number of sample results multiplied by 100. As specified in the PQOs, the goal for completeness for target analytes in each analytical fraction is 90 percent.

Percent completeness is calculated using the following equation:

$$\%C = (T - R)/T \times 100$$

Where:

%C	=	percent completeness
T	=	total number of sample results
R	=	total number of rejected sample results

Completeness is also determined by comparing the planned number of samples per method and matrix as specified in the project planning document, with the number determined above.

Sensitivity is the ability of an analytical method or instrument to discriminate between measurement responses representing different concentrations. This capability is established during the planning phase to meet the DQOs. It is important that calibration requirements, detection limits (DLs), and project-specific LODs and LOQs presented in the work plan are achieved and that target analytes can be detected at concentrations necessary to support the DQOs. In addition, sample results are compared to method blank and field blank results to identify potential effects of laboratory background and field procedures on sensitivity.

The following sections present a review of QC data for each analytical method.

2. Polynuclear Aromatic Hydrocarbons

A total of 58 soil samples were analyzed for PAH by EPA SW-846 Method 8270C-SIM. All PAH data were assessed to be valid with the exception of 17 of the 986 total results, which were rejected based on QC exceedances. This section discusses the QA/QC supporting documentation as defined by the PARCC criteria and evaluated based on the PQOs.

2.1 Precision and Accuracy

2.1.1 Instrument Calibration

Initial and continuing calibration results provide a means of evaluating accuracy within a particular SDG. Relative response factor (RRF), percent relative standard deviation (%RSD), and percent difference (%D) are the three major parameters used to measure the effectiveness of instrument calibration. RRF is a measure of the relative spectral response of an analyte compared to its internal standard. %RSD is an expression of the linearity of instrument response. %D is a comparison of a continuing calibration instrumental response with its initial response. %RSD and %D exceedances suggest routine instrumental anomalies, which typically impact all sample results for the affected compounds.

The relative response factors met the acceptance criteria of 0.05 in the initial and continuing calibration standards.

The relative standard deviation in the initial calibrations and/or %D between the initial calibration mean relative response factors and the continuing calibration relative response factors were within the acceptance criteria of 15 and 20 percent, respectively.

The %Ds in the initial calibration verification were within the acceptance criteria of 20 percent.

2.1.2 Surrogates

As a result of non-compliant surrogate recoveries, 17 non-detected results in sample BA368 were qualified as unusable (R). Additionally, 136 results in samples BA267, BA338, BA341, BA363, BA364, BA367, BA368, and BA369 were qualified as detected estimated (J) and non-detected estimated (UJ) due to non-compliant surrogate recoveries. The details regarding the qualification of results are provided in the data validation reports.

2.1.3 MS/MSD Samples

As a result of non-compliant MS/MSDs, five results for non-compliant RPDs and 32 results for non-compliant %Rs were qualified as detected estimated (J) and non-detected estimated (UJ). The affected compounds were 2-methylnapthalene, acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, fluorene, naphthalene, phenanthrene, and pyrene. The details regarding the qualification of results are provided in the data validation reports.

2.1.4 LCS Samples

As a result of non-compliant LCS/LCSD recoveries, 139 results were qualified as detected estimated (J) and non-detected estimated (UJ). The affected compounds were acenaphthene, benzo(a)anthracene, benzo(a)pyrene, dibenz(a,h)anthracene, fluorene, and pyrene. The details regarding the qualification of results are provided in the data validation reports.

2.1.5 Internal Standards

No data were qualified based on internal standard nonconformances. The recoveries and retention times were evaluated against the acceptance criteria.

2.1.6 Field Duplicate Samples

The field duplicate samples were evaluated for acceptable precision with RPDs for the compounds. The associated data validation narratives provided details regarding criteria exceeded. Sample data were not qualified on the basis of field duplicate precision.

2.1.7 Proficiency Testing Samples

Proficiency testing samples were not performed for the sampling event.

2.1.8 Compound Quantitation and Target Identification

Due to compound quantitation nonconformances (i.e., co-elution of peaks), 29 benzo(b)fluoranthene and benzo(k)fluoranthene detected results in several samples were qualified as detected estimated (J). The details regarding the qualification of results are provided in the data validation reports.

All target compound identifications were found to be acceptable

2.2 REPRESENTATIVENESS

2.2.1 Holding Times

The evaluation of holding times to verify compliance with the method was conducted. All holding times were met.

2.2.2 Blanks

Method blanks were analyzed to evaluate representativeness. The concentration for an individual target compounds in any of the three types of QA/QC blanks were used for data qualification.

If contaminants were detected in a blank, corrective actions were made for the chemical analytical data during data validation. The corrective action consisted of amending the laboratory reported results for organic compounds based on the following criteria. The validation qualifier codes used in the blank summary tables are described below.

- *Results Below or Above the LOQ:* If a sample result for the blank contaminant was less than the LOQ or greater than the sample LOQ and less than 5× the blank value, the sample result for the blank contaminant was amended as a non-detect at the concentration reported in the sample results.
- *No Action:* If a sample result for the blank contaminant was greater than 10× the blank value for common contaminants or 5× the blank value for other contaminants, the result was not amended.

2.2.2.1 METHOD BLANKS

As a result of method blank contamination, one benzo(a)anthracene result was qualified as non-detected (U). The details regarding the qualification of results are provided in the data validation reports.

2.3 COMPARABILITY

The laboratory used standard analytical methods for all of the analyses. In all cases, the method detection limits attained were at or below the reporting limit. Target compounds detected below the reporting limits flagged (J) by the laboratory should be considered estimated. The comparability of the data is regarded as acceptable.

2.4 COMPLETENESS

The completeness level attained for PAH field samples was 98.3 percent. This percentage was calculated as the total number of accepted sample results divided by the total number of sample results multiplied by 100.

2.5 SENSITIVITY

The calibration was evaluated for instrument sensitivity and was determined to be technically acceptable. All laboratory reporting limits met the specified requirements described in the work plan although LOD was elevated for benzo(a)anthracene for one sample due to method blank contamination.

3. Polychlorinated Biphenyls

A total of 20 soil samples were analyzed for PCB as Aroclors by EPA SW-846 Method 8082. All PCB data were assessed to be valid since none of the 140 total results were rejected based on QC exceedances. This section discusses the QA/QC supporting documentation as defined by the PARCC criteria and evaluated based on the PQOs.

3.1 PRECISION AND ACCURACY

3.1.1 Instrument Calibration

Initial and continuing calibration results provide a means of evaluating accuracy within a particular SDG. Percent relative standard deviation (%RSD) and percent difference (%D) are the two major parameters used to measure the effectiveness of instrument calibration. %RSD is an expression of the linearity of instrument response. %D is a comparison of a continuing calibration instrumental response with its initial response. %RSD and %D exceedances suggest more routine instrumental anomalies, which typically impact all sample results for the affected compounds.

Six results were qualified detected estimated (J) and non-detected estimated (UJ). The relative standard deviations in the initial calibrations and/or percent difference between the initial calibration and the continuing calibration concentrations for Aroclor 1016, Aroclor 1221, and Aroclor 1232 were outside the acceptance criteria of 20 and 15 percent, respectively. The affected samples are identified in the data validation reports.

3.1.2 Surrogates

No data were qualified based on surrogate recovery nonconformances. In cases where individual recoveries exceeded criteria, the QC exceedance was judged to have no impact on the data quality and no qualifications were made.

3.1.3 MS/MSD Samples

No data were qualified based on MS/MSD nonconformances. For those SDGs with MS/MSD results, the recoveries were evaluated against the acceptance criteria. In cases where recoveries exceeded criteria, the QC exceedance was judged to have no impact on the data quality and no qualifications were made.

3.1.4 LCS Samples

No data were qualified based on LCS nonconformances. For those SDGs with LCS results, the recoveries were evaluated against the acceptance criteria.

3.1.5 Field Duplicate Samples

The field duplicate samples were evaluated for acceptable precision with RPDs for the compounds. The associated data validation narratives provided details regarding criteria exceeded. Sample data were not qualified on the basis of field duplicate precision.

3.1.6 Proficiency Testing Samples

Proficiency testing samples were not performed for the sampling event.

3.1.7 Compound Quantitation and Target Identification

Due to compound quantitation nonconformances (i.e., %Ds between columns), one Aroclor 1260 result in sample BA245 was qualified as detected estimated (J). The details regarding the qualification of results are provided in the data validation reports.

All target compound identifications were found to be acceptable.

3.2 REPRESENTATIVENESS

3.2.1 Holding Times

The evaluation of holding times to verify compliance with the method was conducted. All holding times were met.

3.2.2 Blanks

As previously discussed in Section 2.2.2, method blanks were analyzed to evaluate representativeness.

3.2.2.1 METHOD BLANKS

No QC issues were associated with the method blanks for this analysis.

3.3 COMPARABILITY

The laboratory used standard analytical methods for all of the analyses. In all cases, the method detection limits attained were at or below the reporting limit. Target compounds detected below the reporting limits flagged (J) by the laboratory should be considered estimated. The comparability of the data is regarded as acceptable.

3.4 COMPLETENESS

The completeness level attained for PCB field samples was 100 percent. This percentage was calculated as the total number of accepted sample results divided by the total number of sample results multiplied by 100.

3.5 SENSITIVITY

The calibration was evaluated for instrument sensitivity and was determined to be technically acceptable. All laboratory LODs and LOQs met the project requirements described in the work plan.

4. Metals

A total of 48 soil samples were analyzed for metals by EPA SW-846 Method 6010B/6020/7471A. All metals data were assessed to be valid since none of the 465 total results were rejected based on QC exceedances. This section discusses the QA/QC supporting documentation as defined by the PARCC criteria and evaluated based on the PQOs.

4.1 PRECISION AND ACCURACY

4.1.1 Instrument Calibration

Initial and continuing calibration verification results provide a means of evaluating accuracy within a particular SDG. Correlation coefficient (r) and percent recovery (%R) are the two major parameters used to measure the effectiveness of instrument calibration. The correlation coefficient indicates the linearity of the calibration curve. %R is used to verify the ongoing calibration acceptability of the analytical system. The most critical of the two calibration parameters, r, has the potential to affect data accuracy across a SDG when it is outside the acceptable QC limits. %R exceedances suggest more routine instrumental anomalies, which typically impact all sample results for the affected analytes.

The correlation coefficients in the initial calibrations and/or percent recoveries in the continuing calibration verifications were within the acceptance criteria of $\geq$ 0.995 and 90-110 percent, respectively.

4.1.2 MS Samples

As a result of non-compliant MS recoveries, 21 results were qualified as detected estimated (J) and non-detected estimated (UJ). The analytes affected were barium, cadmium, and chromium. The details regarding the qualification of results are provided in the data validation reports.

4.1.3 Duplicate (DUP) Samples

No data were qualified based on duplicate nonconformances. For those SDGs with DUP results, the relative percent differences/differences were evaluated against the acceptance criteria. In cases where

RPDs or differences exceeded criteria, the QC exceedance was judged to have no impact on the data quality and no qualifications were made.

4.1.4 LCS Samples

No data were qualified based on LCS nonconformances. For those SDGs with LCS results, the recoveries were evaluated against the acceptance criteria.

4.1.5 ICP Serial Dilution

No data were qualified based on ICP serial dilution nonconformances. All recoveries were evaluated against the acceptance criteria.

4.1.6 ICP Interference Check Sample

As a result of ICP interference check sample exceedances, 16 results were qualified as detected estimated (J) and non-detected estimated (UJ). The analytes affected were arsenic, cadmium, chromium, and silver. The details regarding the qualification of results are provided in the data validation reports.

4.1.7 Field Duplicate Samples

The field duplicate samples were evaluated for acceptable precision with RPDs for the analytes. The associated data validation narratives provided details regarding criteria exceeded. Sample data were not qualified on the basis of field duplicate precision.

4.1.8 Proficiency Testing Samples

Proficiency testing samples were not performed for the sampling event.

4.1.9 Sample Result Verification

All sample results were found to be acceptable.

4.2 REPRESENTATIVENESS

4.2.1 Holding Times

The evaluation of holding times to verify compliance with the method was conducted. All holding times were met.

4.2.2 Blanks

Method blanks were analyzed to evaluate representativeness. The concentration for an individual target compounds in any of the three types of QA/QC blanks were used for data qualification.

If contaminants were detected in a blank, corrective actions were made for the chemical analytical data during data validation. The corrective action consisted of amending the laboratory reported results for organic analytes based on the following criteria. The validation qualifier codes are described below.

- *Results Below or Above the LOQ:* If a sample result for the blank contaminant was less than the LOQ or greater than the sample LOQ and less 5× the method blank value or the highest

applicable calibration blank value, the sample result for the blank contaminant was amended as a non-detect at the concentration reported in the sample results.

- *No Action:* If a sample result for the blank contaminant was greater than 5× the blank value, the result was not amended.

4.2.2.1 METHOD BLANKS

No QC issues were associated with the method blanks for this analysis.

4.3 COMPARABILITY

The laboratory used standard analytical methods for all of the analyses. In all cases, the method detection limits attained were at or below the reporting limit. Target analytes detected below the reporting limits flagged (J) by the laboratory should be considered estimated. The comparability of the data is regarded as acceptable.

4.4 COMPLETENESS

The completeness level attained for metal field samples was 100 percent. This percentage was calculated as the total number of accepted sample results divided by the total number of sample results multiplied by 100.

4.5 SENSITIVITY

The calibration was evaluated for instrument sensitivity and was determined to be technically acceptable. All laboratory LODs and LOQs met the project requirements described in the work plan.

5.0 Variances in Analytical Performance

The laboratory used standard analytical methods for all of the analyses throughout the project. No systematic variances in analytical performance were noted according to the laboratory SOW.

6.0 Summary of PARCC criteria

The validation reports present the PARCC results for all SDGs. Each PARCC criterion is discussed in detail in the following sections.

6.1 PRECISION AND ACCURACY

Precision and accuracy were evaluated using data quality indicators such as MS/MSD, LCS, and surrogates. The precision and accuracy of the data set were considered acceptable after integration of qualification of estimated results as specifically noted in the data validation reports.

6.2 REPRESENTATIVENESS

All samples for each method and matrix were evaluated for holding time compliance. All samples were associated with a method blank in each individual SDG. The representativeness of the project data is considered acceptable after qualification for blank contamination.

6.3 COMPARABILITY

Sampling frequency requirements were met in obtaining duplicates and necessary field blanks. The laboratory used standard analytical methods for their analyses. The analytical results were reported in correct standard units. Holding times, sample preservation, and sample integrity were within QC criteria. The overall comparability is considered acceptable.

6.4 COMPLETENESS

Of the 1591 total analytes reported, 17 of the sample results were rejected. The completeness for all SDGs is as follows:

Parameter/Method	Total Analytes	No. of Rejects	%Completeness
PAHs	986	17	98.3
PCBs	140	0	100
Metals	465	0	100
Total	**1,591**	**17**	**98.9**

The completeness percentage based on rejected data met the 90 percent DQO goal. A less quantifiable loss of data occurred in the application of blank qualifications.

6.5 SENSITIVITY

Sensitivity was achieved by the laboratory to support the DQOs. Calibration concentrations and reporting limits met the project requirements and low level PAH contamination in the method blanks did not affect sensitivity.

Table 1: Validation Sample Table, SDG 42300

Client ID #	Lab ID #	QC Type	Matrix	Date Collected	Mercury (7470A)	PAH (8270C-SIM)	PCBs (8082)
BA268	AP55206		soil	7-30-03	X		
BA269	AP55207		soil	7-30-03	X		
BA270	AP55208		soil	7-30-03	X		
BA271	AP55209		soil	7-30-03	X		
BA272	AP55210		soil	7-30-03	X		
BA273	AP55211		soil	7-30-03	X		
BA274	AP55212		soil	7-30-03	X		
BA275	AP55213		soil	7-30-03	X		
BA276	AP55214		soil	7-30-03	X		
BA277	AP55215		soil	7-30-03	X		
BA278	AP55216		soil	7-31-03	X		
BA279	AP55217		soil	7-31-03	X		
BA280	AP55218		soil	7-31-03	X		
BA281	AP55219		soil	7-31-03	X		
BA282	AP55220		soil	7-31-03	X		
BA283	AP55221		soil	7-31-03	X		
BA284	AP55222		soil	7-31-03	X		
BA285	AP55223		soil	7-31-03	X		
BA286	AP55224		soil	7-31-03	X		
BA287	AP55225		soil	7-31-03	X		
BA245	AP54789		soil	7-25-03			X
BA246	AP54790		soil	7-25-03			X
BA247	AP54791		soil	7-25-03			X
BA248	AP54792		soil	7-25-03			X
BA249	AP54793		soil	7-25-03			X
BA250	AP54794		soil	7-25-03			X
BA251	AP54795		soil	7-25-03			X
BA252	AP54796		soil	7-25-03			X
BA253	AP54797		soil	7-25-03			X
BA254	AP54798		soil	7-25-03			X
BA255	AP54799		soil	7-25-03			X
BA256	AP54800		soil	7-25-03			X
BA257	AP54801		soil	7-25-03			X
BA258	AP54802		soil	7-25-03			X
BA259	AP54803		soil	7-25-03			X
BA260	AP54804		soil	7-25-03			X
BA261	AP54805		soil	7-25-03			X
BA262	AP54806		soil	7-25-03			X
BA263	AP54807		soil	7-25-03			X
BA264	AP54808		soil	7-25-03			X
BA265	AP54809		soil	7-26-03		X	
BA265DL	AP54809DL	DL	soil	7-26-03		X	

Client ID #	Lab ID #	QC Type	Matrix	Date Collected	Mercury (7470A)	PAH (8270C-SIM)	PCBs (8082)
BA266	AP54810		soil	7-26-03		X	
BA266DL	AP54810DL	DL	soil	7-26-03		X	
BA266DL2	AP54810DL2	DL2	soil	7-26-03		X	
BA267	AP54811		soil	7-26-03		X	
BA245MS	AP54789MS	MS	soil	7-25-03			X

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Laboratory QC Samples (Water, Soil)

1. Purpose

This section sets forth the standard operating procedure for identifying the number and type of laboratory quality control (QC) samples that will be analyzed during each contract task order (CTO) associated with the United States Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific. Laboratory QC analyses serve as a check on the precision and accuracy of analytical methods and instrumentation, and the potential contamination that might occur during laboratory sample preparation and analyses. Laboratory QC analyses include blank, surrogate, blank spike, laboratory control sample (LCS), and matrix spike (MS)/matrix spike duplicate (MSD) analyses. These laboratory QC analyses are discussed in general below.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

3.1 Precision

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as a standard deviation, variance, or range, in either absolute or relative terms. Examples of QC measures for precision include laboratory duplicates, laboratory triplicates, and matrix spike/matrix spike duplicates.

3.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias), components which are due to sampling and analytical operations. Examples of QC measures for accuracy include performance evaluation samples, matrix spikes, LCSs, and equipment blanks.

3.3 Matrix

A specific type of medium (e.g., surface water, drinking water), in which the analyte of interest may be contained. Medium is a substance (e.g., air, water, soil), which serves as a carrier of the analytes of interest (EPA 2010).

3.4 METHOD BLANK

An analyte-free matrix (water, soil, etc.) subjected to the entire analytical process to demonstrate that the analytical system itself does not introduce contamination.

3.5 MATRIX SPIKE

A sample prepared by adding a known concentration of a target analyte to an aliquot of a specific homogenized environmental sample for which an independent estimate of the target analyte concentration is available. The MS is accompanied by an independent analysis of the unspiked aliquot of the environmental sample. Spiked samples are used to determine the effect of the matrix on a method's recovery efficiency.

3.6 LABORATORY CONTROL SAMPLES AND BLANK SPIKES

A sample of known composition prepared using reagent-free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is analyzed using the sample preparation, reagents, and analytical methods employed for regular samples.

3.7 SURROGATES

A pure substance with properties that mimic the analyte of interest (organics only). Surrogates are typically brominated, fluorinated, or isotopically labeled compounds unlikely to be found in environmental samples. These analytes are added to samples to evaluate analytical efficiency by measuring recovery.

3.8 INTERNAL STANDARDS

A pure substance added to both samples and laboratory standards at a known concentration with the purpose of providing a basis of comparison in the quantitation of analytes of interest. Internal standards are primarily used to increase the accuracy and precision of analytical methods where the primary source of variability is in sample preparation or sample injection on instrument.

4. Responsibilities

The prime contractor's QA Manager or Technical Director, as well as QC coordinators are responsible for ensuring that sample analytical activities during all CTOs are in compliance with this procedure.

The CTO QC Coordinators and the Laboratory Manager are responsible for identifying instances of non-compliance with this procedure and ensuring that future laboratory analytical activities are in compliance with it.

5. Procedures

Laboratory QC checks include all types of samples specified in the requested analytical methods, such as the analysis of laboratory blank, duplicate, and MS samples. QC requirements are specified in each analytical method and in Appendix B, *Quality Control Requirements*, and Appendix C, *Laboratory Control Sample (LCS) Control Limits and Requirements*, of the *Department of Defense Quality Systems Manual for Environmental Laboratories* Version 5.0 (or most current version)

(DoD QSM). Types of QC samples are discussed in general below. Detailed discussion and minimum QA/QC requirements are presented in the DoD QSM (DoD 2013).

A comprehensive discussion of the minimum number of laboratory QC samples can be found in the *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities* (DoD 2005b). However, additional QA/QC samples may be necessary based on the project quality objectives. Information pertaining to laboratory QC samples shall be documented in Worksheet 28 Laboratory QC Samples Table of the project UFP QAPP-style planning document.

5.1 LABORATORY BLANKS

Laboratory blank samples are analyzed to assess the degree to which laboratory contamination by reagent or method preparation may have affected sample analytical results. At a minimum, one laboratory blank will be analyzed per matrix per analytical method for each batch of at most 20 samples. In evaluating the blank results, all blank data are reviewed to identify any compounds detected in the blanks. The laboratory shall be contacted to discuss detection of analytes in blank samples only in the event of unusual contamination, but not for common laboratory contaminants at low levels. The following compounds are considered to be common laboratory contaminants: acetone, methylene chloride, 2-butanone, and common phthalate esters. The data for samples analyzed during the same time period as the blank are then evaluated to identify the presence of any contaminants found in the blanks. The presence of the blank contaminants found in associated samples is then evaluated to avoid potential misinterpretation of actual sample constituents. Briefly, as discussed in the data validation procedures, any analyte detected above the LOQ in both the sample and the associated blank is qualified as not detected if the sample concentration is less than five times the blank concentration (5× rule). For common laboratory contaminants (methylene chloride, acetone, 2-butanone, and common phthalate esters), a 10× rule applies.

5.2 LABORATORY REPLICATES (DUPLICATES AND TRIPLICATES)

Replicates are analyzed to evaluate the reproducibility, or precision, of the analytical procedures for a given sample. A replicate is two (duplicates) or three (triplicates) representative portions taken from one homogeneous sample by the laboratory and analyzed in the same laboratory (DoD 2005a). One duplicate sample is analyzed for each batch of twenty samples analyzed in a given matrix. Lab triplicates are assigned by the field team and identified on the chain of custody. The identification of a sample for lab triplicate analysis is typically selected from one of the field triplicates to allow for the evaluation of total study error of the sampling and analysis process. Duplicate analyses are normally performed on sample portions analyzed for inorganic constituents. For organic analyses, duplicate analyses are performed on MS samples (Section 5.5 of this procedure).

5.3 SURROGATES

Surrogate compounds must be added to all samples, standards, and blanks for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery. Surrogate compounds to be included for organic analysis are specified in each analytical method.

5.4 Laboratory Control Samples and Blank Spikes

LCSs are used to demonstrate that the laboratory process for sample preparation and analysis is under control.

Analytes selected for spiking of LCSs are usually the same compounds used to spike MS/MSD samples and are representative target compounds. Control limits for LCS recoveries are provided in Appendix C of DoD QSM. If no control limits for LCS recoveries are listed in Appendix C of the DoD QSM for a given analyte, the laboratory's in-house derived control limits should be used.

For wet chemistry methods, a single spike of an appropriate control for each method may be used for LCS analyses (i.e., cyanide, a control standard of sodium cyanide from a source other than that used for calibration may be spiked into water samples and analyzed with the water samples). LCSs should be analyzed at a frequency of one per batch of at most twenty samples analyzed of similar matrix.

5.5 Matrix Spikes/Matrix Spike Duplicates

MS analyses are conducted by the laboratory to assess the accuracy of specific analytical methods and to provide information on the effect of the sample matrix on the analytical methodology. Spike analyses are performed by adding compounds of known concentration to a sample, an unspiked portion of which has previously been analyzed or is concurrently analyzed. The spiked analytes are representative target compounds for each analytical method performed. The spiked sample results are evaluated with the original sample results to evaluate any effects the matrix has on the analysis. One MS is analyzed for each batch of at most 20 samples of similar matrix. Since MS samples only provide information about the specific sample matrix used for the spike, MS analyses should be performed for each type of matrix collected.

For the MSD, a separate aliquot of the sample is separately spiked and analyzed. As discussed in Section 5.2, results of MSD analyses are expressed as a relative percent difference, which is calculated by dividing the difference in concentration between the MSD and the MS sample analyses by the arithmetic mean of their concentrations. One MSD analysis is required for at most each 20 samples of similar matrix.

Acceptance criteria for both the MS and the MSD are based on historic laboratory performance and are laboratory-specific. As a general rule, the acceptance criteria should be no more stringent than the LCS acceptance criteria.

It is important to note that the UFP QAPP Part 2B, QA/QC Compendium: Minimum QA/QC Activities (DoD 2005b) states that for organic analysis, MS and MSDs are not considered a minimum QC activity as long as surrogate spikes properly mimic the analytes of concern and can identify matrix effects. Project quality objectives should be evaluated to determine if organic MS/MSDs are useful for individual projects.

6. Records

Records of QC samples analyzed during ER Program CTO activities will be maintained on laboratory bench sheets, raw data sheets, in the laboratory computerized data system, and on QC summary forms, as requested. Analytical laboratories maintain records in accordance with their quality assurance manual (QAM) as part of performing environmental analytical work under DoD.

Records shall be maintained in accordance with the analytical laboratory subcontract agreement specifications or the laboratory-specific QAM, whichever is more stringent.

7. Health and Safety

Applicable to laboratory personnel only.

8. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/swerffrr/pdf/-qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories*. Version 5.0. Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Environmental Protection Agency, United States (EPA). 2010. Environmental Monitoring and Assessment Program: QA Glossary. November 8. On-line updates available at: http://www.epa.gov/emfjulte/html/pubs/docs/resdocs/qa_terms.html#mm. Accessed 2015.

Procedure I-A-7, *Analytical Data Validation Planning and Coordination.*

9. Attachments

None.

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Field QC Samples (Water, Soil)

1. Purpose

This standard operating procedure describes the number and types of field quality control (QC) samples that will be collected during United States Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific site field work.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the Contract Task Order (CTO) Manager and the Quality Assurance (QA) Manager or Technical Director, as well as QC coordinators responsible for compliance with the procedure. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

3.1 Trip Blank

Trip blanks are samples that originate from organic-free water (e.g., ASTM Type II water, high performance liquid chromatography grade water, etc.) prepared by the laboratory, shipped to the sampling site, and returned to the laboratory with samples to be analyzed for volatile organic compounds (VOCs). Trip blanks are analyzed to assess whether contamination was introduced during sample shipment (DoD 2005a). Trip blanks are prepared using the same sample container (typically a 40 ml VOA vial) as that used to collect field samples.

3.2 Equipment Blank Samples

An equipment blank (i.e., "decontamination rinsate," or "equipment rinsate") sample consists of a sample of water free of measurable contaminants poured over or through decontaminated field sampling equipment that is considered ready to collect or process an additional sample. Equipment blanks are to be collected from non-dedicated sampling equipment to assess the adequacy of the decontamination process.

3.3 Field Blanks

A blank used to provide information about contaminants that may be introduced during sample collection, storage, and transport. It can also be a clean sample carried to the sampling site, exposed to sampling conditions, transported to the laboratory, and treated as an environmental sample.

3.4 FIELD DUPLICATE

A generic term for two field samples taken at the same time in approximately the same location is referred to as a field duplicate. The location of the duplicate (distance and direction from primary sample) should be specified in the project planning documents. They are intended to represent the same population and are taken through all steps of the analytical procedure in an identical manner and provide precision information for the data collection activity. There are two categories of field duplicate samples defined by the collection method: co-located field duplicates and subsample field duplicates. Co-located field duplicates are two or more independent samples collected from side-by-side locations at the same point in time and space so as to be considered identical. Co-located samples are collected from adjacent locations or liners (e.g., laterally or vertically, in separate containers), or water samples collected from the same well at the same time that have not been homogenized. Subsample field duplicates samples are obtained from one sample collection at one sample location.

3.5 FIELD REPLICATES

Two or more field replicates are used with incremental sampling approaches to statistically evaluate the sampling precision or error for each decision unit (DU). The location of the replicates (distance and direction from primary sample) and the number of DUs with replicates should be specified in the project planning documents. Increments for replicate samples are collected from completely separate locations (i.e., separate systematic random or stratified random grid). Triplicate samples (i.e., primary incremental sample plus two replicates) are required for incremental sampling and are more useful than just duplicates for statistical evaluation. The replicate samples are collected, prepared, and analyzed in the same manner as carried out for the primary sample.

3.6 TEMPERATURE INDICATORS (BLANKS)

A temperature indicator sample is often referred to as a temperature blank, but it is not analyzed nor does it measure introduced contamination. It may be a small sample bottle or VOA vial filled with distilled water that is placed in each shipping container to evaluate if samples were adequately cooled during sample shipment.

3.7 SOURCE WATER

Source water is water free from measurable contaminants that is used as the final decontamination rinse water.

4. Responsibilities

The prime contractor CTO Manager and QA Manager or Technical Director are responsible for ensuring that field QC samples are collected and analyzed according to this procedure. The CTO Manager is responsible for ensuring that all personnel involved in sampling or testing shall have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

The prime contractor QC Coordinator is responsible for determining the QC sample requirements.

The Laboratory Manager is responsible for ensuring that field QC samples are analyzed according to the specifications of the project statement of work and the analytical methods used.

The Field Manager is responsible for ensuring that all project field staff follow these procedures.

Field sampling personnel are responsible for the implementation of this procedure.

5. Procedures

Field QC checks may include submission of trip blank, equipment blank, field blank, duplicate, triplicate, and temperature indicator (blank) samples to the laboratory. Types of field QC samples are discussed in general below. Table III-B-1 identifies the minimum frequency at which field QC samples should be collected, with the actual frequency to be determined by the individual project needs. For additional information on field QC frequency, see the State of Hawaii Department of Health 2009 *Technical Guidance Manual for the Implementation of the Hawaii State Contingency Plan.*

A comprehensive discussion of the minimum types and numbers of field QC samples can be found in the *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities* (DoD 2005).

Table III-B-1: Field QC Samples per Sampling Event

Type of Sample	Minimum QC Sample Frequency	
	Metals	Organic
Trip blank (for volatiles only)	N/A	1/analytical method/cooler
Equipment blank	5%	5%
Field blank	1/decontamination water source/event [a]/for all analytes	
Field replicates [b]	10%	10%
Temperature Indicator (blank)	1/shipping container	

% percent
N/A not applicable
[a] A sampling event is considered to be from the time sampling personnel arrive at a site until they leave for more than a week. The use of controlled-lot source water makes one sample per lot, rather than per event, an option.
[b] To the extent practical, field replicates should be collected from the same locations as the samples designated for a laboratory matrix spike/matrix spike duplicate (organic analysis) where applicable, or from the sample used as a laboratory duplicate (inorganic analysis).

5.1 Trip Blanks

The laboratory prepares trip blanks using organic-free water, and then sends them to the field. The laboratory shall place trip blanks in sample coolers prior to transport to the site so that they accompany the samples throughout the sample collection/handling/transport process. Once prepared, trip blanks should not be opened until they reach the laboratory. One set of two 40-milliliter vials per volatile analysis forms a trip blank and accompanies each cooler containing samples to be analyzed for volatiles. Trip blanks are only analyzed for volatiles. Results of trip blank analyses are used to assess whether samples have been contaminated by volatiles during sample handling and transport to the laboratory.

Trip blanks are not typically associated with tissue samples; however, project-specific quality objectives shall determine if trip blanks for tissue samples are required.

5.2 EQUIPMENT BLANK SAMPLES

Collect equipment blank samples by pumping the source water over and/or through the decontaminated sampling equipment. Collect this runoff water into the sample containers directly or with the use of a funnel, if necessary. The source water may be pumped or poured by tipping the jug of water upside down over the equipment. Results of equipment blank samples are used to evaluate whether equipment decontamination was effective.

At a minimum, equipment blank samples should be collected at a rate of 5 percent of the total samples planned for collection for each sampling technique used. This rate may be adjusted depending on the nature of the investigation (site inspection, remedial investigation, remedial site evaluation, long-term monitoring) and the associated project quality objectives (PQOs). Equipment blank samples will be analyzed for the same parameters as the samples collected with that particular equipment. If analytes pertinent to the project are found in the equipment blanks, the frequency of equipment blank samples may be increased after decontamination procedures have been modified to further evaluate the effectiveness of the decontamination procedure.

When disposable or dedicated sampling equipment is used, equipment blank samples do not need to be collected.

Sampling devices (e.g., gloved hands, dip nets, or traps) used for collection of tissue samples are generally non-intrusive into the organisms collected, so equipment blank samples will not be collected as long as the devices have been properly cleaned following Procedure I-F, *Equipment Decontamination*, and appear clean.

5.3 FIELD BLANKS

Field blanks, consisting of samples of the source water used as the final decontamination rinse water, will be collected on site by field personnel by pouring the source water into sample containers and then analyzed to assess whether contaminants may have been introduced during sample collection, storage, and transport.

The final decontamination rinse water source (the field blank source water) and equipment blank source water should all be from the same purified water source. Tap water used for steam cleaning augers or used in the initial decontamination buckets need not be collected and analyzed as a field blank since augers typically do not touch the actual samples and the final decontamination rinse water should be from a purified source.

Field blanks should be collected at a minimum frequency of one per sampling event per each source of water. A sampling event is considered to be from the time sampling personnel arrive at a site until they leave for more than a week. Field blanks will be analyzed for the same parameters as the samples collected during the period that the water sources are being used for decontamination. Additional field blanks may be required based on PQOs.

5.4 FIELD DUPLICATES

Field duplicates consist of either co-located or subsampled samples. Field duplicates for ground water and surface water samples are generally considered to be co-located samples. Soil duplicate samples may be homogenized and subsampled in the field (or at the laboratory) to form an original

and duplicate sample, or may be an additional volume of sample collected in a separate sample container to form a co-located sample.

The interpretation of co-located duplicate data may be more complex than subsample duplicate data because of the number of variables associated with the results of this type of duplicate sample. Duplicate soil samples for VOC analysis shall always be co-located (i.e., not homogenized or otherwise processed or subsampled). Duplicates will be analyzed for the same analytical parameters as their associated original sample. Collection of both co-located and subsampled versions of the same sample may be performed to aid in approximating sampling and analysis error.

Field duplicates for biological tissue samples will consist of subsamples of the original sample. Twice the required volume of organisms for one sample will be collected and placed into one food-grade, self-sealing bag. The sample will later be homogenized in the laboratory and subsampled, producing an original and a duplicate sample. Tissue duplicate samples will be analyzed for the same analytical parameters as their associated original samples.

5.5 Field Replicates

Field replicates are completely separate incremental replicate samples (collected from a set of systematic random or stratified random locations within the DU that are different from those used for the primary incremental samples). A different random starting location is determined for each replicate collected in the selected DU. Field replicates are typically collected in sets of three (the primary sample and two replicate samples) to produce a triplicate.

Replicate sample increments are collected from the same sampling grid established through the DU for the primary incremental sample, though at different systematic random locations than initially used. The replicate increments should not be collected from the same points or co-located with those used for the primary incremental sample. Replicate samples are sent to the laboratory as "blind" samples, meaning the laboratory does not know they represent replicate samples of the primary incremental sample.

5.6 Temperature Indicators (Blanks)

Temperature indicators (blanks) may be prepared in the lab or field by filling a small sample bottle or VOA vial with distilled water and sealing the container. One temperature indicator sample should be placed in each sample cooler or shipping container. Upon arrival at the laboratory, the temperature of the bottle is measured to determine if samples were adequately cooled during the shipment.

6. Records

Records of QC samples analyzed during ER Program CTO activities will be maintained on laboratory bench sheets, raw data sheets, in the laboratory computerized data system, and on QC summary forms, as requested. Analytical laboratories maintain records in accordance with their quality assurance manual (QAM) as part of performing environmental analytical work under DoD. Records shall be maintained in accordance with the analytical laboratory subcontract agreement specifications or the laboratory-specific QAM, whichever is more stringent.

7. Health and Safety

Field personnel shall perform work in accordance with the current (or as contractually obligated) United States Army Corps of Engineers Safety and Health Requirements Manual EM-385-1-1 (USACE 2008) and site-specific health and safety plan.

8. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/swerffrr/pdf/-qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories*. Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual*. OPNAV Instruction 5090.1D. 10 January.

United States Army Corps of Engineers (USACE). 2008. *Consolidated Safety and Health Requirements Manual*. EM-385-1-1. Includes Changes 1–7. 13 July 2012.

Procedure I-F, *Equipment Decontamination*.

Procedure III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody*.

9. Attachments

None.

Logbooks

1. Purpose

This standard operating procedure describes the activities and responsibilities pertaining to the identification, use, and control of logbooks and associated field data records for use by United States Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific personnel.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan Appendix A. Section 1.4 *Field Documentation SOPs* (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the Contract Task Order (CTO) Manager and the Quality Assurance Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

3.1 LOGBOOK

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person assigned responsibility for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 DATA FORM

A data form is a predetermined format used for recording field data that may become, by reference, a part of the logbook (e.g., soil boring logs, trenching logs, surface soil sampling logs, groundwater sample logs, and well construction logs are data forms).

4. Responsibilities

The prime contractor CTO Manager or delegate is responsible for determining which team members shall record information in field logbooks and for obtaining and maintaining control of the required logbooks. The CTO Manager shall review the field logbook on at least a monthly basis. The CTO Manager or designee is responsible for reviewing logbook entries to determine compliance with this procedure and to ensure that the entries meet the project requirements.

A knowledgeable individual such as the Field Manager, CTO Manager, or quality control (QC) Supervisor shall perform a technical review of each logbook at a frequency commensurate with the level of activity (weekly is suggested, or, at a minimum, monthly). Document these reviews by the

dated signature of the reviewer on the last page or page immediately following the material reviewed.

The Field Manager is responsible for ensuring that all project field staff follow these procedures and that the logbook is completed properly and daily. The Field Manager is also responsible for submitting copies to the CTO Manager, who is responsible for filing them and submitting a copy to the Navy (if required by the CTO Statement of Work).

The logbook user is responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. The logbook user is also responsible for safeguarding the logbook while having custody of it.

Field personnel are responsible for the implementation of this procedure.

All NAVFAC Pacific ER Program field personnel are responsible for complying with Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

5. Procedure

The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct the applicable events. Store the logbook in a clean location and use it only when outer gloves used for personal protective equipment (PPE) have been removed.

Individual data forms may be generated to provide systematic data collection documentation. Entries on these forms shall meet the same requirements as entries in the logbook and shall be referenced in the applicable logbook entry. Individual data forms shall reference the applicable logbook and page number. At a minimum, include names of all samples collected in the logbook even if they are recorded elsewhere.

Enter field descriptions and observations into the logbook, as described in Attachment III-D-1, using indelible black ink.

Typical information to be entered includes the following:

- Dates (month/day/year) and times (military) of all onsite activities and entries made in logbooks/forms
- Site name, and description
- Site location by longitude and latitude, if known
- Weather conditions, including estimated temperature and relative humidity
- Fieldwork documentation, including site entry and exit times
- Descriptions of, and rationale for, approved deviations from the work plan or field sampling plan
- Field instrumentation readings
- Names, job functions, and organizational affiliations of personnel on-site

- Photograph references
- Site sketches and diagrams made on-site
- Identification and description of sample morphology, collection locations and sample numbers as described in Procedure I-A-8, *Sample Naming*
- Sample collection information, including dates (month/day/year) and times (military) of sample collections, sample collection methods and devices, station location numbers, sample collection depths/heights, sample preservation information, sample pH (if applicable), analysis requested (analytical groups), etc., as well as chain-of-custody (COC) information such as sample identification numbers cross-referenced to COC sample numbers
- Sample naming convention
- Field QC sample information
- Site observations, field descriptions, equipment used, and field activities accomplished to reconstruct field operations
- Meeting information
- Important times and dates of telephone conversations, correspondence, or deliverables
- Field calculations
- PPE level
- Calibration records
- Contractor and subcontractor information (address, names of personnel, job functions, organizational affiliations, contract number, contract name, and work assignment number)
- Equipment decontamination procedures and effectiveness
- Laboratories receiving samples and shipping information, such as carrier, shipment time, number of sample containers shipped, and analyses requested
- User signatures

The logbook shall reference data maintained in other logs, forms, etc. Correct entry errors by drawing a single line through the incorrect entry, then initialing and dating this change. Enter an explanation for the correction if the correction is more than for a mistake.

At least at the end of each day, the person making the entry shall sign or initial each entry or group of entries.

Enter logbook page numbers on each page to facilitate identification of photocopies.

If a person's initials are used for identification, or if uncommon acronyms are used, identify these on a page at the beginning of the logbook.

At least weekly and preferably daily, the preparer shall photocopy (or scan) and retain the pages completed during that session for backup. This will prevent loss of a large amount of information if the logbook is lost.

6. Records

Retain the field logbook as a permanent project record. If a particular CTO requires submittal of photocopies of logbooks, perform this as required.

7. Health and Safety

Store the logbook in a clean location to keep it clean and use it only when outer gloves used for PPE have been removed.

8. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual*. OPNAV Instruction 5090.1D. 10 January.

Procedure I-A-8, *Sample Naming.*

9. Attachments

Attachment III-D-1: Description of Logbook Entries

Attachment III-D-1
Description of Logbook Entries

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Logbook entries shall be consistent with Section A.1.4 *Field Documentation SOPs* of the UFP-QAPP Manual (DoD 2005) and contain the following information, as applicable, for each activity recorded. Some of these details may be entered on data forms, as described previously.

Name of Activity	For example, Asbestos Bulk Sampling, Charcoal Canister Sampling, Aquifer Testing.
Task Team Members and Equipment	Name all members on the field team involved in the specified activity. List equipment used by serial number or other unique identification, including calibration information.
Activity Location	Indicate location of sampling area as indicated in the field sampling plan.
Weather	Indicate general weather and precipitation conditions.
Level of PPE	Record the level of PPE (e.g., Level D).
Methods	Indicate method or procedure number employed for the activity.
Sample Numbers	Indicate the unique numbers associated with the physical samples. Identify QC samples.
Sample Type and Volume	Indicate the medium, container type, preservative, and the volume for each sample.
Time and Date	Record the time and date when the activity was performed (e.g., 0830/08/OCT/89). Use the 24-hour clock for recording the time and two digits for recording the day of the month and the year.
Analyses	Indicate the appropriate code for analyses to be performed on each sample, as specified in the WP.
Field Measurements	Indicate measurements and field instrument readings taken during the activity.
Chain of Custody and Distribution	Indicate chain-of-custody for each sample collected and indicate to whom the samples are transferred and the destination.
References	If appropriate, indicate references to other logs or forms, drawings, or photographs employed in the activity.
Narrative (including time and location)	Create a factual, chronological record of the team's activities throughout the day including the time and location of each activity. Include descriptions of general problems encountered and their resolution. Provide the names and affiliations of non-field team personnel who visit the site, request changes in activity, impact the work schedule, request information, or observe team activities. Record any visual or other observations relevant to the activity, the contamination source, or the sample itself. It should be emphasized that logbook entries are for recording data and chronologies of events. The logbook author must include observations and descriptive notations, taking care to be objective and recording no opinions or subjective comments unless appropriate.
Recorded by	Include the signature of the individual responsible for the entries contained in the logbook and referenced forms.
Checked by	Include the signature of the individual who performs the review of the completed entries.

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Record Keeping, Sample Labeling, and Chain-Of-Custody

1. Purpose

The purpose of this standard operating procedure is to establish standard protocols for all United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific field personnel for use in maintaining field and sampling activity records, writing sample logs, labeling samples, ensuring that proper sample custody procedures are used, and completing chain-of-custody/analytical request forms.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

3.1 LOGBOOK

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person responsible for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 CHAIN-OF-CUSTODY

Chain-of-custody (COC) is documentation of the process of custody control. Custody control includes possession of a sample from the time of its collection in the field to its receipt by the analytical laboratory, and through analysis and storage prior to disposal.

4. Responsibilities

The prime contractor CTO Manager is responsible for determining which team members shall record information in the field logbook and for checking sample logbooks and COC forms to ensure compliance with these procedures. The CTO Manager shall review COC forms on a monthly basis at a minimum.

The prime contractor CTO Manager and QA Manager or Technical Director are responsible for evaluating project compliance with the Project Procedures Manual. The QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

The Laboratory Project Manager or Sample Control Department Manager is responsible for reporting any sample documentation or COC problems to the CTO Manager or CTO Laboratory Coordinator within 24 hours of sample receipt.

The Field Manager is responsible for ensuring that all field personnel follow these procedures. The CTO Laboratory Coordinator is responsible for verifying that the COC/analytical request forms have been completed properly and match the sampling and analytical plan. The CTO Manager or CTO Laboratory Coordinator is responsible for notifying the laboratory, data managers, and data validators in writing if analytical request changes are required as a corrective action. These small changes are different from change orders, which involve changes to the scope of the subcontract with the laboratory and must be made in accordance with a respective contract (e.g., Comprehensive Long-Term Environmental Action Navy, remedial action contract).

NAVFAC Pacific ER Program field personnel are responsible for following these procedures while conducting sampling activities. Field personnel are responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. All NAVFAC Pacific ER Program field personnel are responsible for complying with Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

5. Procedures

This procedure provides standards for documenting field activities, labeling the samples, documenting sample custody, and completing COC/analytical request forms. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented.

5.1 Record Keeping

The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct each day's events. Field logs such as soil boring logs and groundwater sampling logs will also be used. These procedures are described in Procedure III-D, *Logbooks*.

5.2 Sample Labeling

Affix a sample label with adhesive backing to each individual sample container with the exception of pre-tared containers. Record the following information with a waterproof marker (ballpoint pen for containers for volatile analyses) on each label:

- Project name or number (optional)
- COC sample number
- Date and time of collection
- Sampler's initials
- Matrix (optional)
- Sample preservatives (if applicable)

- Analysis to be performed on sample (This shall be identified by the method number or name identified in the subcontract with the laboratory)
- Indicate if sample is to be used as the matrix spike (MS)/matrix spike duplicate (MSD) or laboratory triplicate sample

With the exception of sample containers with pre-tared labels, place clear tape over each label (preferably prior to sampling) to prevent the labels from tearing off, falling off, or being smeared, and to prevent loss of information on the label.

These labels may be obtained from the analytical laboratory or printed from a computer file onto adhesive labels.

For volatile soil organic analyses (VOA), labels are not to be affixed to vials that are pre-tared by the laboratory. Instead, on each of the VOA vials in the sample set (typically three per sample), mark the sample COC Sample identification (ID) on the vial in ballpoint pen. Then wrap the vials together in bubble wrap and place one sample label on the bubble wrap and cover with tape. It is imperative that the COC Sample ID be clearly marked on each vial as this will help prevent laboratory error if the vials are inadvertently separated after removal from the bubble wrap.

5.3 CUSTODY PROCEDURES

For samples intended for chemical analysis, sample custody procedures shall be followed through collection, transfer, analysis, and disposal to ensure that the integrity of the samples is maintained. Maintain custody of samples in accordance with the U.S. Environmental Protection Agency (EPA) COC guidelines prescribed in U.S. Environmental Protection Agency (EPA) *NEIC Policies and Procedures*, National Enforcement Investigations Center, Denver, Colorado, revised August 1991 (EPA 1978); EPA *RCRA Ground Water Monitoring Technical Enforcement Guidance Document* (TEGD), *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA OSWER Directive 9355 3-01) (EPA 1988, Appendix 2 of the *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports* (Cal/EPA 1988), and *Test Methods for Evaluating Solid Waste* (EPA 2007). A description of sample custody procedures is provided below.

5.3.1 Sample Collection Custody Procedures

According to the EPA guidelines, a sample is considered to be in custody if one of the following conditions is met:

- It is in one's actual physical possession or view
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal)
- It is retained in a secured area with restricted access
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal

Place custody seals on sample containers (on bubble wrap for pre-tared containers) immediately after sample collection and on shipping coolers if the cooler is to be removed from the sampler's custody.

Place custody seals in such a manner that they must be broken to open the containers or coolers. Label the custody seals with the following information:

- Sampler's name or initials
- Date and time that the sample/cooler was sealed

These seals are designed to enable detection of sample tampering. An example of a custody seal is shown in Attachment III-E-1.

Field personnel shall also log individual samples onto COC forms (carbon copy or computer generated) when a sample is collected or just prior to shipping. These forms may also serve as the request for analyses. Procedures for completing these forms are discussed in Section 5.4, indicating sample identification number, matrix, date and time of collection, number of containers, analytical methods to be performed on the sample, and preservatives added (if any). The samplers will also sign the COC form signifying that they were the personnel who collected the samples. The COC form shall accompany the samples from the field to the laboratory. When a cooler is ready for shipment to the analytical laboratory, the person delivering the samples for transport will sign and indicate the date and time on the accompanying COC form. One copy of the COC form will be retained by the sampler and the remaining copies of the COC form shall be placed inside a self-sealing bag and taped to the inside of the cooler. Each cooler must be associated with a unique COC form. Whenever a transfer of custody takes place, both parties shall sign and date the accompanying carbon copy COC forms, and the individual relinquishing the samples shall retain a copy of each form. One exception is when the samples are shipped; the delivery service personnel will not sign or receive a copy because they do not open the coolers. The laboratory shall attach copies of the completed COC forms to the reports containing the results of the analytical tests. An example COC form is provided in Attachment III-E-2.

5.3.2 Laboratory Custody Procedures

The following custody procedures are to be followed by an independent laboratory receiving samples for chemical analysis; the procedures in their Naval Facilities Engineering and Expeditionary Warfare Center-evaluated Laboratory Quality Assurance Plan must follow these same procedures. A designated sample custodian shall take custody of all samples upon their arrival at the analytical laboratory. The custodian shall inspect all sample labels and COC forms to ensure that the information is consistent, and that each is properly completed. The custodian will also measure the temperature of the temperature blank in the coolers upon arrival using either a National Institute for Standards and Technology calibrated thermometer or an infra-red temperature gun. The custodian shall note the condition of the samples including:

- If the samples show signs of damage or tampering
- If the containers are broken or leaking
- If headspace is present in sample vials
- Proper preservation of samples (made by pH measurement, except volatile organic compounds (VOCs) and purgeable total petroleum hydrocarbons (TPH) and temperature). The pH of VOC and purgeable TPH samples will be checked by the laboratory analyst after the sample aliquot has been removed from the vial for analysis.

- If any sample holding times have been exceeded

All of the above information shall be documented on a sample receipt sheet by the custodian.

Discrepancies or improper preservation shall be noted by the laboratory as an out-of-control event and shall be documented on an out-of-control form with corrective action taken. The out-of-control form shall be signed and dated by the sample control custodian and any other persons responsible for corrective action. An example of an out-of-control form is included as Attachment III-E-4.

The custodian shall then assign a unique laboratory number to each sample and distribute the samples to secured storage areas maintained at 4 degrees Celsius (soil samples for VOC analysis are to be stored in a frozen state until analysis). The unique laboratory number for each sample, the COC sample number, the client name, date and time received, analysis due date, and storage shall also be manually logged onto a sample receipt record and later entered into the laboratory's computerized data management system. The custodian shall sign the shipping bill and maintain a copy.

Laboratory personnel shall be responsible for the care and custody of samples from the time of their receipt at the laboratory through their exhaustion or disposal. Samples should be logged in and out on internal laboratory COC forms each time they are removed from storage for extraction or analysis.

5.4 COMPLETING COC/ANALYTICAL REQUEST FORMS

COC form/analytical request form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

Attachment III-E-2 is an example of a generic COC/analytical request form that may be used by field personnel. Multiple copies may be tailored to each project so that much of the information described below need not be handwritten each time. Attachment III-E-3 is an example of a completed site-specific COC/analytical request form, with box numbers identified and discussed in text below.

Box 1 *Project Manager:* This name shall be the name that will appear on the report. Do not write the name of the Project Coordinator or point of contact for the project instead of the CTO manager.

Project Name: Write the project name as it is to appear on the report.

Project Number: Write the project number as it is to appear on the report. It shall include the project number and task number. Also include the laboratory subcontract number.

Box 2 *Bill to:* List the name and address of the person/company to bill only if it is not in the subcontract with the laboratory.

Box 3 *Sample Disposal Instructions:* These instructions will be stated in the Master Service Agreement or each CTO statement of work with each laboratory.

Shipment Method: State the method of shipment (e.g., hand carry; air courier via FED EX, AIR BORNE, or DHL).

Comment: This area shall be used by the field team to communicate observations, potential hazards, or limitations that may have occurred in the field or additional information regarding analysis (e.g., a specific metals list, samples expected to contain high analyte concentrations).

Box 4 *Cooler Number:* This will be written on the inside or outside of the cooler and shall be included on the COC. Some laboratories attach this number to the trip blank identification, which helps track volatile organic analysis samples. If a number is not on the cooler, field personnel shall assign a number, write it on the cooler, and write it on the COC.

QC Level: Enter the reporting/QC requirements (e.g., Full Data Package, Summary Data Package).

Turn around time (TAT): TAT will be determined by a sample delivery group (SDG), which may be formed over a 14-day period, not to exceed 20 samples. Once the SDG has been completed, standard TAT is 21 calendar days from receipt of the last sample in the SDG. Entering NORMAL or STANDARD in this field will be acceptable. If quicker TAT is required, it shall be in the subcontract with the laboratory and reiterated on each COC to remind the laboratory.

Box 5 *Type of containers:* Write the type of container used (e.g., 1 liter glass amber, for a given parameter in that column).

Preservatives: Field personnel must indicate on the COC the correct preservative used for the analysis requested. Indicate the pH of the sample (if tested) in case there are buffering conditions found in the sample matrix.

Box 6 *COC sample number:* This is typically a five-character alpha-numeric identifier used by the contractor to identify samples. The use of this identifier is important since the labs are restricted to the number of characters they are able to use. See Procedure I-A-8, *Sample Naming*.

Description (sample identification): This name will be determined by the location and description of the sample, as described in Procedure I-A-8, *Sample Naming*. This sample identification should not be submitted to the laboratory, but should be left blank. If a computer COC version is used, the sample identification can be input, but printed with this block black. A cross-referenced list of COC Sample Number and sample identification must be maintained separately.

Identify if sample requires laboratory subsampling.

Date Collected: Record the collection date to track the holding time of the sample. Note: For trip blanks, record the date it was placed in company with samples.

Time Collected: When collecting samples, record the time the sample is first collected. Use of the 24-hour military clock will avoid a.m. or p.m. designations (e.g., 1815 instead of 6:15 p.m.). Record local time; the laboratory is responsible for calculating holding times to local time.

Lab Identification: This is for laboratory use only.

Box 7 *Matrix and QC:* Identify the matrix (e.g., water, soil, air, tissue, fresh water sediment, marine sediment, or product). If a sample is expected to contain high analyte concentrations (e.g., a tank bottom sludge or distinct product layer), notify the laboratory in the comment section. Mark an "X" for the sample(s) that have extra volume for laboratory QC matrix spike/matrix spike duplicate (MS/MSD) or laboratory triplicate purposes. The sample provided for MS/MSD purposes is usually a field duplicate.

Box 8 *Analytical Parameters:* Enter the parameter by descriptor and the method number desired (e.g. benzene, toluene, ethylbenzene, and xylenes 8260B, polynuclear aromatic hydrocarbons 8270C, etc.). Whenever practicable, list the parameters as they appear in the laboratory subcontract to maintain consistency and avoid confusion.

If the COC does not have a specific box for number of sample containers, use the boxes below the analytical parameter, to indicate the number of containers collected for each parameter.

Box 9 *Sampler's Signature:* The person who collected samples must sign here.

Relinquished By: The person who turned over the custody of the samples to a second party other than an express mail carrier, such as FEDEX, must sign here.

Received By: Typically, a representative of the receiving laboratory signs here. Or, a field crew member who delivered the samples in person from the field to the laboratory might sign here. A courier, such as Federal Express, does not sign here because they do not open the coolers. It must also be used by the prime contracting laboratory when samples are to be sent to a subcontractor.

Relinquished By: In the case of subcontracting, the primary laboratory will sign the Relinquished By space and fill out an additional COC to accompany the samples being subcontracted.

Received By (Laboratory): This space is for the final destination (e.g., at a subcontracted laboratory).

Box 10 *Lab Number and Questions:* This box is to be filled in by the laboratory only.

Box 11 *Control Number:* This number is the "COC" followed by the first contractor identification number in that cooler, or contained on that COC. This control number must be unique (i.e., never used twice). Record the date the COC is completed. It should be the same date the samples are collected.

Box 12 *Total No. of Containers/row:* Sum the number of containers in that row.

Box 13 *Total No. of Containers/column:* Sum the number of containers in that column. Because COC forms contain different formats depending on who produced the form, not all of the information listed in items 1 to 13 may be recorded; however, as much of this information as possible shall be included.

COC forms tailored to each CTO can be drafted and printed onto multi-ply forms. This eliminates the need to rewrite the analytical methods column headers each time. It also eliminates the need to write the project manager, name, and number; QC Level; TAT; and the same general comments each time.

Complete one COC form per cooler. Whenever possible, place all volatile organic analyte vials into one cooler in order to reduce the number of trip blanks. Complete all sections and be sure to sign and date the COC form. One copy of the COC form must remain with the field personnel.

6. Records

The COC/analytical request form shall be faxed or e-mailed to the CTO Laboratory Coordinator for verification of accuracy. Following the completion of sampling activities, the sample logbook and COC forms will be transmitted to the CTO Manager for storage in project files. The data validators shall receive a copy also. The original COC/analytical request form shall be submitted by the laboratory along with the data delivered. Any changes to the analytical requests that are required shall be made in writing to the laboratory. A copy of this written change shall be sent to the data validators and placed in the project files. The reason for the change shall be included in the project files so that recurring problems can be easily identified.

7. Health and Safety

Not applicable.

8. References

California Environmental Protection Agency (Cal/EPA). 1988. *Technical Guidance Manual, Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports.* Solid Waste Disposal Program, Hydrogeology Section, Land Disposal Branch, Division of Water Quality, State Water Resources Control Board. August.

Department of Defense, United States (DoD). 2005a. Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/swerffrr/pdf/-qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual.* OPNAV Instruction 5090.1D. 10 January.

Environmental Protection Agency, United States (EPA). 1978. *NEIC Policies and Procedures.* EPA-330/9-78-001-R. Revised August 1991. National Enforcement Investigation Center. Denver. May.

———. 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA.* Interim Final. EPA/540/G-89/004. Office of Emergency and Remedial Response. October.

———. 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Revision 6. Office of Solid Waste. November. On-line updates at: http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm.

Procedure I-A-8, *Sample Naming.*

Procedure III-D, *Logbooks.*

9. Attachments

Attachment III-E-1, Chain-of-Custody Seal

Attachment III-E-2, Generic Chain-of-Custody/Analytical Request Form

Attachment III-E-3, Sample Completed Chain-of-Custody

Attachment III-E-4, Sample Out-of-Control Form

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Attachment III-E-1
Chain-of-Custody Seal

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CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	
Company Name (808) XXX-XXXX	
Sampler's Name/Initials:_____	Date: _____ Time: _____

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Attachment III-E-2
Generic Chain-of-Custody/Analytical Request Form

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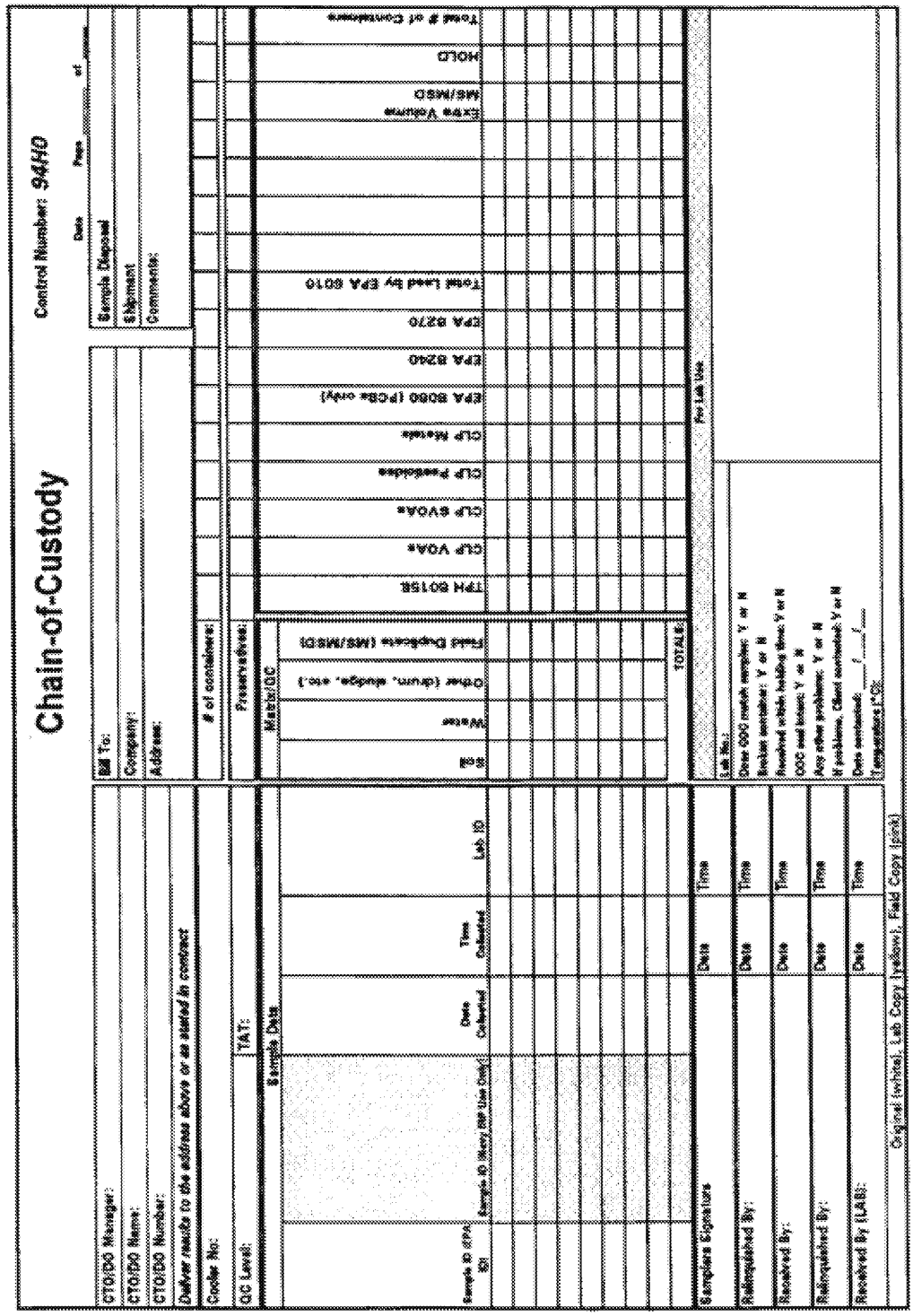

Generic Chain-of-Custody/Analytical Request Form

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Attachment III-E-3
Sample Completed Chain-of-Custody

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Chain-of-Custody

(11) Control Number: 96H0HC205

Date 9 / 3 / 96 Page 1 of 1

(1)

CTO/DO Manager: Joe Smith
CTO/DO Name: Former Navy Landfill
CTO/DO Number: CTO 0250
Deliver results to the address above or as stated in contract

(2)

Bill To: CLEAN/RAC Contractor
Company: company name
Address: Oahu, Hawaii

(3)

Sample Disposal: by lab
Shipment Method: Express Courier
Comments: PACDIV Level D, Measure Cooler Temperature at Lab

(4)

Cooler No: 413
QC Level: PACDIV Level D
TAT: Normal - per contract

(5)

container # (water):	1	2	2	1	2	1											2		
Preservatives:	HCL	HCL			HNO3														

Sample Data

(6)

Sample ID (EPA ID)	Sample ID (Navy IRP Use Only)	Date Collected	Time Collected	Lab ID	(7) Matrix/QC: Soil	Water	Other (drum, sludge, etc.)	Field Duplicate (MS/MSD)	(8) TPH 8015B	CLP VOAs	CLP SVOAs	CLP Pesticides	CLP Metals	EPA 8080 (PCBs only)	EPA 8240	EPA 8270	Total Lead by EPA 6010					Extra Volume MS/MSD	HOLD	(12) Total # of Containers
HC205		9/6/96	9:35		X				X	X	X	X	X	X										1
HC206		9/6/96	9:50		X				X	X	X	X	X	X										1
HC207		9/6/96	10:15		X				X	X	X	X	X	X										1
HC208		9/6/96	10:25		X				X	X	X	X	X	X										1
HC209		9/6/96	10:45		X				X	X	X	X	X	X										1
HC210		9/6/96	10:55			X		X		X														2
HC211		9/6/96	12:50			X			X	X	X	X	X	X										9
								TOTALS:	6	9	7	6	7	6										16

(13)

(9)

Samplers Signature	Date	Time
Relinquished By:	Date	Time
Received By:	Date	Time
Relinquished By:	Date	Time
Received By (LAB):	Date	Time

(10) For Lab Use

Lab No.:
Does COC match samples: Y or N
Broken container: Y or N
Received within holding time: Y or N
COC seal intact: Y or N
Any other problems: Y or N
If problems, Client contacted: Y or N
Date contacted: ___/___/___
Temperature (°C):

Original (white), Lab Copy (yellow), Field Copy (pink)

Sample Completed Chain-of-Custody

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Attachment III-E-4
Sample Out-of-Control Form

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OUT OF CONTROL FORM	Status	Date	Initial
	Noted OOC		
	Submit for CA*		
	Resubmit for CA*		
	Completed		

Date Recognized:	By:
Dated Occurred:	Matrix
Parameter (Test Code):	Method:
Analyst:	Supervisor:

Samples Affected (List by Accession AND Sample No.)

1. Type of Event (Check all that apply)		2. Corrective Action (CA)* (Check all that apply)	
	Calibration Corr. Coefficient <0.995		Repeat calibration
	%RSD>20%		Made new standards
	Blank >MDL		Reran analysis
	Does not meet criteria:		Sample(s) redigested and rerun
	Spike		Sample(s) reextracted and rerun
	Duplicate		Recalculated
	LCS		Cleaned system
	Calibration Verification		Ran standard additions
	Standard Additions		Notified
	MS/MSD		Other (please explain)
	BS/BSD		
	Surrogate Recovery		
	Calculations Error		

	Holding Times Missed	
	Other (Please explain	Comments:

3. Results of Corrective Action	
	Return to Control (indicated with)
	Corrective Actions Not Successful - DATA IS TO BE FLAGGED with _____.

Analyst:	Date:
Supervisor:	Date:
QA Department:	Date:

Sample Handling, Storage, and Shipping

1. Purpose

This standard operating procedure sets forth the methods for use by the United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific personnel engaged in handling, storing, and transporting samples.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

None.

4. Responsibilities

The prime contractor CTO Manager and the Laboratory Project Manager are responsible for identifying instances of non-compliance with this procedure and ensuring that future sample transport activities are in compliance with this procedure.

The Field Manager is responsible for ensuring that all samples are shipped according to this procedure.

Field personnel are responsible for the implementation of this procedure.

The QA Manager or Technical Director is responsible for ensuring that sample handling, storage, and transport activities conducted during all CTOs are in compliance with this procedure.

All field personnel are responsible for complying with Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

5. Procedures

5.1 Handling and Storage

Immediately following collection, label all samples according to Procedure III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody*. In addition, when more than one volatile organic analyte

(VOA) vial is used to collect one sample, the chain-of-custody (COC) identification (ID) will be written on the VOA vials (even pre-tared vials) with a ball point pen for that sample. The lids of the containers shall not be sealed with duct tape, but should be covered with custody seals (except pre-tared containers which should have the custody seal placed on the outside of the protective bubble wrap). Wrap glass sample containers on the sides, tops, and bottoms with bubble wrap or other appropriate padding to prevent breakage during transport. When collecting three VOA vials per sample, it is acceptable to wrap all three vials together and store in one plastic bag. Store all glass containers for water samples in an upright position, never stacked or placed on their sides. Samples will be maintained as close to 4 degrees Celsius (°C) as possible from the time of collection through transport to the analytical laboratory, using refrigerators and/or freezers when appropriate. Place all containers into self-sealing bags and into an insulated cooler with wet ice while still in the field. Samples should occupy the lower portion of the cooler, while the ice should occupy the upper portion. Place an absorbent material (e.g., proper absorbent cloth material) on the bottom of the cooler to contain liquids in case of spillage. Ship samples as soon after collection as possible to allow the laboratory to meet holding times for analyses. Check with the laboratory for operating/sample receipt hours prior to all traditional and non-traditional holidays to ensure sample shipment will be received. When not shipping samples directly upon field collection, store samples in a refrigerator or freezer (never freeze water samples) until shipped to the laboratory.

5.2 PACKING

Each cooler must contain a temperature blank (small plastic bottle with sterile water) to confirm cooler temperature upon receipt at the laboratory. Water samples can be used as such, but it is best to include a designated temperature blank bottle, typically supplied by the laboratory with the coolers.

One trip blank must be included in each cooler containing samples for volatile analysis (e.g., volatile organic compounds, total petroleum hydrocarbons-gasoline range organics.

Cooler must be lined completely in ice at the bottom and all four sides. After confirming all project samples are accounted for and labeled correctly, place samples in cooler. Record sample IDs on cooler-specific COC(s). Pack glass containers for water samples in an upright position, never stacked or placed on their sides. Fill all empty space between sample containers with bubble wrap or other appropriate material (not Styrofoam). Place a layer of ice on top of samples and fill all empty space between ice and cooler lid with bubble wrap or other appropriate material.

Place laboratory copies of completed COC(s), and soil permit if applicable, into resealable bag and tape to underside of cooler lid.

5.3 SHIPPING

Follow all appropriate U.S. Department of Transportation regulations (e.g., 49 Code of Federal Regulations [CFR], Parts 171-179) for shipment of air, soil, water, and other samples. Elements of these procedures are summarized below.

5.3.1 Hazardous Materials Shipment

Field personnel must state whether any sample is suspected to be a hazardous material. A sample should be assumed to be hazardous unless enough evidence exists to indicate it is non-hazardous. If not suspected to be hazardous, shipments may be made as described in the Section 5.3.3 for non-hazardous materials. If hazardous, follow the procedures summarized below.

Any substance or material that is capable of posing an unreasonable risk to life, health, or property when transported is classified as hazardous. Perform hazardous materials identification by checking the list of dangerous goods for that particular mode of transportation. If not on that list, materials can be classified by checking the Hazardous Materials Table (49 CFR 172.102 including Appendix A) or by determining if the material meets the definition of any hazard class or division (49 CFR Part 173), as listed in Attachment III-F-2.

All persons shipping hazardous materials must be properly trained in the appropriate regulations, as required by HM-126F, Training for Safe Transportation of Hazardous Materials (49 CFR HM-126F Subpart H). The training covers loading, unloading, handling, storing, and transporting of hazardous materials, as well as emergency preparedness in the case of accidents. Carriers, such as commercial couriers, must also be trained. Modes of shipment include air, highway, rail, and water.

When shipping hazardous materials, including bulk chemicals or samples suspected of being hazardous, the proper shipping papers (49 CFR 172 Subpart C), package marking (49 CFR 172 Subpart D), labeling (49 CFR 172 Subpart E), placarding (49 CFR 172 Subpart F, generally for carriers), and packaging must be used. Attachment III-F-1 shows an example of proper package markings. Refer to a copy of 49 CFR each time hazardous materials/potentially hazardous samples are shipped.

According to Section 2.7 of the International Air Transport Association Dangerous Goods Regulations publication, very small quantities of certain dangerous goods may be transported without certain marking and documentation requirements as described in 49 CFR Part 172. However, other labeling and packing requirements must still be followed. Attachment III-F-2 shows the volume or weight for different classes of substances. A "Dangerous Goods in Excepted Quantities" label must be completed and attached to the associated shipping cooler (Attachment III-F-3). Certain dangerous goods are not allowed on certain airlines in any quantity.

As stated in item 4 of Attachment III-F-4, the Hazardous Materials Regulations do not apply to hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4), and sodium hydroxide (NaOH) added to water samples if their pH or percentage by weight criteria are met. Hazardous Materials Regulations also do not apply to methanol (MeOH) for soil samples if the percentage by weight criterion is met. These samples may be shipped as non-hazardous materials as discussed below.

5.3.2 Non-hazardous Materials Shipment

If the samples are suspected to be non-hazardous based on previous site sample results, field screening results, or visual observations, if applicable, then samples may be shipped as non-hazardous.

If preservatives (HCl, HNO_3, H_2SO_4, NaOH, or MeOH) are used, ensure their individual pH or percentage by weight criteria, as shown in item 4 of Attachment III-F-4, are met to continue shipping as non-hazardous samples.

When a cooler is ready for shipment to the laboratory, place the receiving laboratory address on the top of the cooler, place chain-of-custody seals on the coolers as discussed in Procedure III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody*, place soil permit labels on top if applicable, and seal the cooler with waterproof tape.

5.3.3 Shipments from Outside the Continental United States

Shipment of sample coolers to the continental U.S. from locations outside the continental U.S. is controlled by the U.S. Department of Agriculture (USDA) and is subject to their inspection and regulation. A "USDA Soil Import Permit" is required to prove that the receiving analytical laboratory is certified by the USDA to receive and properly dispose of soil. In addition, all sample coolers must be inspected by a USDA representative, affixed with a label indicating that the coolers contain environmental samples, and accompanied by shipping forms stamped by the USDA inspector prior to shipment. In addition, the U.S. Customs Service must clear samples shipped from U.S. territorial possessions or foreign countries upon entry into the U.S. As long as the commercial invoice is properly completed (see below), shipments typically pass through U.S. Customs Service without the need to open coolers for inspection.

In Hawaii, soil sample shipments are typically brought to the courier at the airport where the courier contacts a USDA representative to make an inspection. Alternatively, the contractor may enter into an agreement with the USDA to ship soil samples. In this way, the USDA does not need to inspect each soil sample shipment. If the contractor maintains a Domestic Soil Permit, place the permit label and the soil origination label (Attachment III-F-9) on the top of the cooler. Place a copy of the receiving laboratory's soil permit with the COC inside the cooler. Confirm custody seals were placed on each container (Section 5.1) to ensure proper chain-of-custody control in the event coolers are opened for inspection.

In Guam, shipments can be dropped off directly to the Federal Express branch or to the courier at the airport. Alternatively, the courier can pick up shipments at each site provided that arrangements have been made regarding pickup time and location. USDA inspections occur outside of Guam. The laboratory's soil permit shall be placed with the COC inside the cooler, and the soil origination label (see Attachment III-F-9) should be placed on top of the cooler.

The USDA does not need to inspect water sample shipments.

Completion and use of proper paperwork will, in most cases, minimize or eliminate the need for the USDA and U.S. Customs Service to inspect the contents. Attachment III-F-5 shows an example of how paperwork may be placed on the outside of coolers for non-hazardous materials. For hazardous materials, refer to Section 5.3.1.

In summary, tape the paperwork listed below to the outside of the coolers to assist sample shipments. If a shipment is made up of multiple pieces (e.g., more than one cooler), the paperwork need only be attached to one cooler, provided that the courier agrees. All other coolers in the shipment need only be taped and have address and COC seals affixed.

1. **Courier Shipping Form & Commercial Invoice**. See Attachment III-F-6, and Attachment III-F-7 for examples of the information to be included on the commercial invoice for soil and water. Place the courier shipping form and commercial invoice inside a clear, plastic, adhesive-backed pouch that adheres to the package (typically supplied by the courier) and place it on the cooler lid as shown in Attachment III-F-5.
2. **Soil Import Permit (soil only)**. See Attachment III-F-8 and Attachment III-F-9 for examples of the soil import permit and soil samples restricted entry labels. The laboratory shall supply these documents prior to mobilization. The USDA in Hawaii often does stop

shipments of soil without these documents. Staple together the 2 inch × 2 inch USDA label (described below), and soil import permit, and place them inside a clear plastic pouch. The courier typically supplies the clear, plastic, adhesive-backed pouches that adhere to the package.

Placing one restricted entry label as shown in Attachment III-F-5 (covered with clear packing tape) and one stapled to the actual permit is suggested.

The USDA does not control water samples, so the requirements for soil listed above do not apply.

3. **Chain-of-Custody Seals**. The laboratory should supply the seals. CTO personnel must sign and date these. At least two seals should be placed in such a manner that they stick to both the cooler lid and body. Placing the seals over the tape (as shown in Attachment III-F-5), then covering it with clear packing tape is suggested. This prevents the seal from coming loose and enables detection of tampering.
4. **Address Label**. Affix a label stating the destination (laboratory address) of each cooler.
5. **Special Requirements for Hazardous Materials**. See Section 5.3.1.

Upon receipt of sample coolers at the laboratory, the sample custodian shall inspect the sample containers as discussed in Procedure III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody*. The samples shall then be either immediately extracted and/or analyzed, or stored in a refrigerated storage area until they are removed for extraction and/or analysis. Whenever the samples are not being extracted or analyzed, they shall be returned to refrigerated storage.

6. Records

Maintain records as required by implementing these procedures.

7. Health and Safety

Personnel shall perform work in accordance with the current (or as contractually obligated) United States Army Corps of Engineers Safety and Health Requirements Manual EM-385-1-1 (USACE 2012) and site-specific health and safety plan.

8. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/swerffrr/pdf/-qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual*. OPNAV Instruction 5090.1D. 10 January.

United States Army Corps of Engineers (USACE). 2008. *Consolidated Safety and Health Requirements Manual.* EM-385-1-1. Includes Changes 1–7. 13 July 2012.

Procedure III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody.*

9. Attachments

Attachment III-F-1: Example Hazardous Materials Package Marking

Attachment III-F-2: Packing Groups

Attachment III-F-3: Label for Dangerous Goods in Excepted Quantities

Attachment III-F-4: SW-846 Preservative Exception

Attachment III-F-5: Non-Hazardous Material Cooler Marking Figure for Shipment From Outside The Continental United States

Attachment III-F-6: Commercial Invoice – Soil

Attachment III-F-7: Commercial Invoice – Water

Attachment III-F-8: Soil Import Permit

Attachment III-F-9: Soil Samples Restricted Entry Labels

Attachment III-F-1
Example Hazardous Material Package Marking

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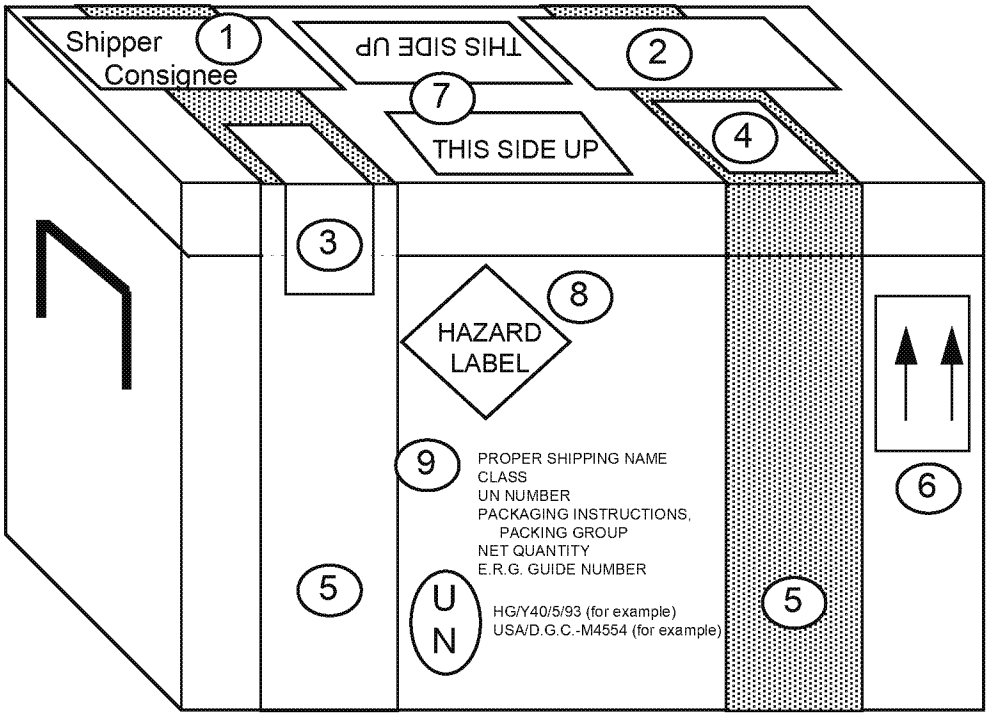

1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**
7. **THIS SIDE UP STICKERS**
8. **HAZARD LABEL**
9. **HAZARDOUS MATERIAL INFORMATION**
10. **PACKAGE SPECIFICATIONS**

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Attachment III-F-2
Packing Groups

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PACKING GROUP OF THE SUBSTANCE	PACKING GROUP 1		PACKING GROUP II		PACKING GROUP III	
CLASS or DIVISION of PRIMARY or SUBSIDIARY RISK	Packagings		Packagings		Packagings	
	Inner	Outer	Inner	Outer	Inner	Outer
1: Explosives	---------- Forbidden (Note A) ----------					
2.1: Flammable Gas	---------- Forbidden (Note B) ----------					
2.2: Non-Flammable, non-toxic gas	---------- See Notes A and B ----------					
2.3: Toxic gas	---------- Forbidden (Note A) ----------					
3. Flammable liquid	30 mL	300 mL	30 mL	500 mL	30 mL	1 L
4.1 Self-reactive substances	Forbidden		Forbidden		Forbidden	
4.1: Other flammable solids	Forbidden		30 g	500 g	30 g	1 kg
4.2: Pyrophoric substances	Forbidden		Not Applicable		Not Applicable	
4.2 Spontaneously combustible substances	Not Applicable		30 g	500 g	30 g	1 kg
4.3: Water reactive substances	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.1: Oxidizers	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.2: Organic peroxides (Note C)	See Note A		30 g or 30 mL	500 g or 250 mL	Not Applicable	
6.1: Poisons - Inhalation toxicity	Forbidden		1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - oral toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1: Poisons - dermal toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.2: Infectious substances	---------- Forbidden (Note A) ----------					
7: Radioactive material (Note D)	---------- Forbidden (Note A) ----------					
8: Corrosive materials	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
9: Magnetized materials	---------- Forbidden (Note A) ----------					
9: Other miscellaneous materials (Note E)	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L

Note A: *Packing groups are not used for this class or division.*
Note B: *For inner packagings, the quantity contained in receptacle with a water capacity of 30 mL. For outer packagings, the sum of the water capacities of all the inner packagings contained must not exceed 1 L.*
Note C: *Applies only to Organic Peroxides when contained in a chemical kit, first aid kit or polyester resin kit.*
Note D: *See 6.1.4.1, 6.1.4.2 and 6.2.1.1 through 6.2.1.7, radioactive material in excepted packages.*
Note E: *For substances in Class 9 for which no packing group is indicated in the List of Dangerous Goods, Packing Group II quantities must be used.*

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Attachment III-F-3
Label for Dangerous Goods in Excepted Quantities

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Attachment III-F-4
SW-846 Preservative Exception

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Measurement	Vol. Req. (mL)	Container[2]	Preservative [3,4]	Holding Time[5]
MBAS	250	P,G	Cool, 4°C	48 Hours
NTA	50	P,G	Cool, 4°C	24 Hours

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-82 (1976) Method D-3370.

2. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

3. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

4. When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. for the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentration of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

6. Should only be used in the presence of residual chlorine.

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Attachment III-F-5
Non-Hazardous Material Cooler Marking Figure for Shipment from outside the Continental United States

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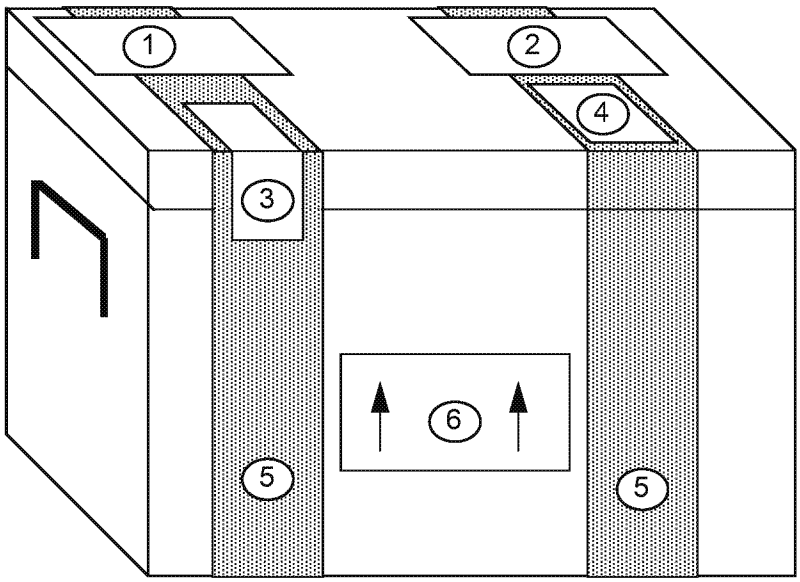

1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**

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Attachment III-F-6
Commercial Invoice – Soil

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DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <CTO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	

INTERNATIONAL AIR WAYBILL NO.

(NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Soil samples for labora analysis only				$1.00	$3.00
	TOTAL NO. OF PKGS.					TOTAL WEIGHT		TOTAL INVOICE VALUE
	3							$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.
DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.
I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT
SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden	Joe Smith	1/1/94
Name/Title	Signature	Date

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Attachment III-F-7
Commercial Invoice – Water

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DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <CTO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Rece <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	

INTERNATIONAL AIR WAYBILL NO.

(NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Water samples for labo analysis only				$1.00	$3.00
	TOTAL NO. OF PKGS.					TOTAL WEIGHT		TOTAL INVOICE VALUE
	3							$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.
DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.
I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT
SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Name/Title	Signature	Date
Joe Smith, Ogden	Joe Smith	1/1/94

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Attachment III-F-8
Soil Import Permit

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Soil Permit

Permit Number: S-52299

UNITED STATES DEPARTMENT OF AGRICULTURE

Animal and Plant Health Inspection Service

Plant Protection and Quarantine

Issued To: Columbia Analytical Services
(Lee Wolf)
1317 S. 13th Avenue
Kelso, Washington 98626

TELEPHONE: (360) 577-7222

Under the authority of the Federal Plant Pest Act of May 23, 1957, permission is hereby granted to the facility/individual named above subject to the following conditions:

1. Valid for shipments of soil not heat treated at the port of entry, only if a compliance agreement (PPQ Form 519) has been completed and signed. Compliance Agreements and Soil permits are non-transferable. If you hold a Soil Permit and you leave your present employer or company, you must notify your local USDA office promptly.
2. To be shipped in sturdy, leakproof, containers.
3. To be released without treatment at the port of entry.
4. To be used only for analysis and only in the facility of the permittee at Columbia Analytical Services, located in Kelso, Washington.
5. No use of soil for growing purposes is authorized, including the isolation or culture of organisms imported in soil.
6. All unconsumed soil, containers, and effluent is to be autoclaved, incinerated, or heat treated by the permittee at the conclusion of the project as approved and prescribed by Plant Protection and Quarantine.
7. This permit authorizes shipments from all foreign sources, including Guam, Hawaii, Puerto Rico, and the U.S. Virgin Islands through any U.S. port of entry.

JUNE 30, 2006
Expiration Date

Deborah M. Knott
Approving Official DEBORAH M. KNOTT

WARNING: Any alteration, forgery, or unauthorized use of this Federal form is subject to civil penalties of up to $250,000 (7 U.S.C. s 7734(b)) or punishable by a fine of not more than $10,000, or imprisonment of not more than 5 years, or both (18 U.S.C. s 1001).

PPQ FORM 525B (8/94)

Pt. 1 - PERMITTEE

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Attachment III-F-9
Soil Samples Restricted Entry Label and Soil Origin Label

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U.S. DEPARTMENT OF AGRICULTURE

ANIMAL AND PLANT HEALTH INSPECTION SERVICE

PLANT PROTECTION AND QUARANTINE

HYATTSVILLE, MARYLAND 20782

SOIL SAMPLES

RESTRICTED ENTRY

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550 *Edition of 12/77 may be used*

(JAN 83)

Soil Samples Restricted Entry Label

SOIL ENCLOSED

Origin of Soil _____

Soil Origin Label

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