ENVIRONMENTAL WORK INSTRUCTION 3EN2.1

From: Environmental Restoration Product Line Coordinator
To: All Environmental Restoration Personnel and Contractors

Subj: CHEMICAL DATA VALIDATION
Ref: (a) DOD 2019. General Data Validation Guidelines, Revision 1, November.

(b) DOD 2020. Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS. Environmental Data Quality Workgroup. May.

(c) DOD 2020. Data Validation Guidelines Module 2: Data Validation Procedure for Metals by ICP-OES. Environmental Data Quality Workgroup. May.

(d) DOD 2020. Data Validation Guidelines Module 3: Data Validation Procedure for Per-nd Polyfluoroalkyl Substances Analysis by QSM Table B-15. May.

(e) DOD 2020. Data Validation Guidelines Module 4: Data Validation Procedure for Organic Analysis by GC. March.

(f) USEPA 2020. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, November.

(g) USEPA 2020. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, November.

(h) USEPA 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G-4. February.

Encl: (1) Data Validation Guidelines for Chemical Analysis of Environmental Samples
(2) Process Flow Chart – Chemical Data Validation

1. PURPOSE: The purpose of the chemical data validation work instruction is to standardize the approach for determination of chemical data validation practices at NAVFAC SW to satisfy project-specific Data Quality Objectives (DQOs) and provide consistency and defensibility of analytical laboratory data.

2. CANCELLATION: EWI#1 3EN2.1 dated 28 November 2001.

3. BACKGROUND: The chemical data validation process consists of a systematic assessment and verification of data quality through independent review. To be independent, validation must be performed by individuals who are not associated with the collection and analysis of samples or the interpretation of sample data.

Data sets are assessed for completeness, compliance with analytical method procedures, and comparison to measurement performance criteria as established in the Sampling and Analysis Plan (SAP).

4. APPLICABILITY: The work instruction on chemical data validation is applicable to all Environmental Restoration Program (ERP) projects within the Naval Facilities Engineering Systems Command Southwest area of responsibility which are funded by the Environmental Restoration, Navy (ER,N) or the Base Realignment and Closure (BRAC) accounts.

5. PROCEDURE: Chemical data validation should consider each type of data, the relationship to the entire data set, and the adequacy of the data to fulfill the DQOs of the sampling event or project. The following procedures are required:

a. Select the level or combination of levels that appropriately support decision-making for your project; refer to Table 1 of enclosure (1) for guidance on the different stages of data validation and their application.

b. Specify the stage of data validation selected in the SAP.

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# ENCLOSURE 1

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ACRONYMS / ABBREVIATIONS

| | |
|---|---|
| CCV | Continuing Calibration Verification |
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act |
| CLP | Contract Laboratory Program |
| DQOs | Data Quality Objectives |
| EPA | Environmental Protection Agency |
| GC/MS | Gas Chromatography/Mass Spectrometry |
| ICV | Initial Calibration Verification |
| IRCDQM | Installation Restoration Chemical Data Quality Manual |
| IS | Internal Standard |
| LCS | Laboratory Control Sample |
| MS/MSD | Matrix Spike/Matrix Spike Duplicate |
| NAVFAC SW | Naval Facilities Engineering Systems Command, Southwest |
| NPL | National Priorities List |
| PCBs | Polychlorinated Biphenyls |
| QAO | Quality Assurance Officer |
| QAPP | Quality Assurance Project Plan |
| QA/QC | Quality Assurance/Quality Control |
| QC | Quality Control |
| RCRA | Resource Conservation and Recovery Act |
| RI/FS | Remedial Investigation/Feasibility Study |
| SAP | Sampling and Analysis Plan |
| SI | Site Inspection |
| SVOCs | Semivolatile Organic Compounds |
| USEPA | United States Environmental Protection Agency |
| VOCs | Volatile Organic Compounds |
| WP | Work Plan |

## 1.0 INTRODUCTION

The process of data validation for laboratory analysis of environmental samples and associated data determines the compliance of the analytical data with established method performance criteria and the specified analytical method data quality objectives (DQOs) of the project. Data validation provides an assessment of data quality and defines the accomplishment of analytical quality control (QC) requirements and project DQOs. This process then provides the basis for the scientific and legal defensibility of the analytical data.

This document is intended for use only as a guidance. Project-specific strategies, as determined by the Project Team, should always supersede the guidance of this document. The data validation strategy deemed most appropriate to facilitate the DQOs and goals of the project investigation must be clearly presented in the project SAP. The Quality Assurance Officer (QAO) will assess the data validation strategy as part of the SAP review and approval process.

This technical guidance was developed to standardize the data validation practices to satisfy project-specific DQOs and provide consistency and defensibility of analytical laboratory data. Specifically, this document is intended to provide guidance to Remedial Project Managers (RPMs) to answer the question of "how much" data should be subjected to the stages of data validation established in support of Department of Defense (DOD) projects. There is no formal guidance that defines the percentage of a total data set that must be subjected to review and validation to meet DQOs and other project requirements. Ultimately, the data validation strategy is a determination made by the Project Team based on investigation-specific considerations. Section 3.0 of this document provides guidance and it is based upon the selection of the data validation "strategy" that may be most appropriate to a particular investigation. A data validation strategy is defined in terms of the percentage of the data set subjected to the validation procedures for the specified stage of data validation (as discussed in Section 2.0).

The data validation process consists of a systematic assessment and verification of data quality through independent review. Validation must be performed by individuals who are not associated with the collection and analysis of samples, interpretation of sample data, or with any decision making process within the scope of the particular investigation. Data validation is not to be performed by the analytical laboratory. Laboratories performing EPA approved methods may attach defined "data flags" under guidance from the laboratory Quality Assurance/Quality Control (QA/QC) protocols. Environmental laboratory data generated in support of Environmental Restoration, Navy (ER,N) or the Base Realignment and Closure (BRAC) investigations shall be validated consistent with references (a) through (g) when applicable.

Data validation is not to be confused with data usability assessment. While data validation provides a systematic process for evaluating analytical uncertainty associated with field and laboratory QC samples relative to performance criteria specified in the SAP, the data usability

assessment addresses the final data disposition based on other considerations, including field sampling processes and project-specific DQOs. Data qualified with an "X" during the validation process (i.e., serious quality control deficiencies) are further evaluated during the data usability assessment to determine whether to accept or reject the qualified data.

## 2.0 STAGES OF DATA VALIDATION

In accordance with reference (h), the USEPA seven-step Data Quality Objectives (DQO) Process is employed to define project-specific DQOs which help determine the QC requirements for field and laboratory analysis, data deliverables, and the type of data review or level of data validation to be performed.

The validation guidelines stages include criteria used in evaluating data from EPA-approved analytical methods primarily from applicable SW-846 methods. Consistent with reference (a), the data may undergo different stages of data validation (Stage 1 through Stage 4) or a combination of these stages depending on project-specific requirements. For methods not yet accepted by EPA, data validation procedures should emulate those outlined in references (f) and (g), and must be specified in the project Sampling and Analysis Plan (SAP) for the appropriate review and approval. Mobile laboratory analyses should be performed at a level of quality control emulating stationary laboratory analysis to ensure data is of sufficient quality to adequately support project requirements. If mobile laboratory analyses are intended only for screening purposes, it should be clearly stated in the project SAP along with a description of the process intended for evaluating the data.

Field screening, process monitoring, progress sampling and waste characterization data may be non-quantitative or semi-quantitative and do not undergo a formal validation. These data must be subjected to a systematic review process using set criteria for data evaluation. The data review process must be specified in the project SAP, and should address data editing, screening, and verification. The data review also determines that instrument calibrations and blanks are within QC criteria. Procedures must also be provided for flagging samples associated with calibrations or blanks which are out of QC criteria. If field screening tests are designed to determine the presence or absence of the target compound, then the evaluation process should examine factors affecting gross interferences in target analyte detection as well as instrument stability. If possible, QC samples such as matrix spikes and duplicates may be included in the screening approach to support field decisions.

The following includes the four stages of data validation procedures as defined in reference (a).

### 2.1 Stages of Data Validation

**Stage 1:** General completeness and verification check to include field sample IDs and target analytes against the chain of custody forms; sample conditions upon arrival; sample preservation requirements; holding times, concentrations and units for limits of detection and

quantitation; trip blanks; field blanks, equipment blanks, and field duplicates (if not submitted blind) frequency and field quality control.

**Stage 2A:** Stage 1 validation plus evaluation of preparatory batch QC results: method blanks, laboratory control samples, matrix spikes, laboratory duplicates (LCSD, MSD, DUP), surrogates (organics), serial dilutions, post digestion spikes (as appropriate to the method), and any preparatory batch cleanup QC to assure project requirements for analyte spike list, frequency, and quality control limits are met.

**Stage 2B:** Stage 2A validation plus evaluation of instrument-related QC results including Instrument Performance Samples: Tunes, breakdown standard check results, peak tailing factors (if applicable), instrument initial calibration summaries (including response factors and any regression summaries), initial calibration verification and continuing calibration verification summaries, internal standards, initial and continuing calibration blank summaries, confirmation of positive results for second column or detector including percent difference between the two analytical concentrations that are greater than the detection limit, and

**Stage 3:** Stage 2B validation plus re-quantification and recalculation of selected samples (i.e., target analytes quantitated from appropriate internal standards) and instrument QC: Appropriate selection of curve fit type, weighting factors, and with or without forcing through zero, continuing calibration verifications and blanks, and percent ratios of tunes and performance checks including calculation of DDT/Endrin breakdown and column peak tailing, and preparatory batch QC results (such as spike percent recoveries and serial dilution percent differences) from instrument response. Instrument response data are required to perform re-quantification and recalculation.

**Stage 4:** Stage 3 validation plus qualitative review of non-detected, detected, and tentatively identified compounds (TICs) from instrument outputs: Chromatograms are checked for peak integration (10% of automated integration and 100% of manual integrations (MI) where chromatograms from before and after MI are examined for cause and justification), baseline, and interferences; mass spectra are checked for minimum signal to noise, qualitative ion mass presence, ion abundances; retention times or relative retention times are within method requirements for analyte identification. Raw data quantitation reports, chromatograms, mass spectra, instrument background corrections, and interference corrections are required to perform review of the instrument outputs.

The extent of data validation that can be performed will be dependent upon the required type of laboratory data deliverable. Successive stages of data validation require more comprehensive data deliverables from the laboratory.

## 3.0 DATA VALIDATION STRATEGY

The guidance for the determination of the data validation needs for a particular project is partly based upon selection of the appropriate validation strategy. A data validation strategy is defined in terms of the type(s) of data validation to be employed (i.e., 10% Stage 4 and 90% Stage 2B) and the percentage of the data set to be validated, as a whole or by each type (stage) of validation.

Each data validation strategy corresponds to a set of considerations that include the status of the site on the National Priorities List (NPL) and potential for a human health risk or ecological risk evaluations in the study. The primary study component considered here is that of risk assessment (human health or ecological), although other study components may influence the choice of data validation strategy for a particular investigation. For example, similar to other analyte groups, if the investigation involves sampling and analyses of Per-and Polyfluoroalkyl Substances (PFAS) in drinking water, that would be an important consideration in the selection of the data validation strategy.

Table 1 depicts potential data validation strategies and the associated considerations for choice of the strategy most appropriate to a given investigation.

Most projects may undergo a combination of these validation stages (i.e., 10% Stage 4 and 90% Stage 2B). The ten percent portion of the data set for Stage 4 validation should consist of both routine and QC samples. In addition, selection of the ten percent may focus on the relative importance of data in the context of the entire project or a single sampling event. Emphasis may be given to a particular analysis parameter (e.g., VOCs, specific target analytes), analytical method, sample type or matrix, or sampling location.

**Table 1. Potential Considerations for Choice of Data Validation Strategy.**

| Validation Strategy | Intended Use | Risk Assessment | Site Type |
|---|---|---|---|
| 10% Stage 4 and 90% Stage 2B* | Investigations and Confirmations on ERP projects | Yes | Non-NPL |
| 20% Stage 4 and 80% Stage 2B* | Investigations involving Drinking Water | Yes | Non-NPL |
| 20% Stage 4 and 80% Stage2B* | Investigations and Confirmations on ERP Projects | Yes | NPL |
| No Formal Data Validation Required | Field Screening Process Monitoring Progress Sampling Waste characterization | No | All |

*- Depending on project-specific DQOs, Stage 3 may replace Stage 2B. Stage 3 incorporates the review of raw data and would equate to a more stringent data validation strategy.

## 4.0 REFERENCES

**DOD 2019.** General Data Validation Guidelines, Revision 1, Environmental Data Quality Workgroup. November 2019.

**DOD 2020.** Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS. Environmental Data Quality Workgroup. May 2020.

**DOD 2020.** Data Validation Guidelines Module 2: Data Validation Procedure for Metals by ICP-OES. Environmental Data Quality Workgroup. May 2020.

**DOD 2020.** Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM 5.3 Table B-15. May 2020.

**DOD 2020.** Data Validation Guidelines Module 4: Data Validation Procedure for Organic Analysis by GC. March 2020.

**USEPA 2020.** Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537. November 2018.

**USEPA 2020.** USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, November 2020.

**USEPA 2020.** USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, November 2020.

**USEPA 2006.** Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G-4. February 2006.

# ENCLOSURE 2

**Process Name: CHEMICAL DATA VALIDATION**
**Date: April 2022**