

6.0 CALIBRATION PROCEDURES AND FREQUENCY

6.0.0.1. Instrument calibration is necessary for accurate quantitation, and establishes the dynamic range of an instrument or piece of equipment. Calibration criteria are instrument-, method-, or equipment-specific. The following paragraphs describe the general requirements for instrument or equipment calibration.

6.1 FIELD INSTRUMENT CALIBRATION

6.1.0.1. Field equipment or instruments shall be calibrated in accordance with the manufacturer's directions and expected field conditions and calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of resulting data can be assessed. Calibration procedures for commonly used field equipment or instruments are defined in SOP 2 in Appendix I of this QAPP. This SOP may be included by reference or as an attachment to the project-specific work plans. Calibration procedures for field equipment or instruments not included in SOP 2 shall be included in the project-specific work plans. Any deviations to SOP 2 shall also be described in the project-specific work plans. A log of maintenance activities for all Hill AFB owned equipment shall be maintained by the contractor for two years after the general contract expires and shall be submitted annually to the Hill AFB Operations and Maintenance Project Manager.

6.2 LABORATORY INSTRUMENT CALIBRATION PROCEDURES FOR DEFINITIVE DATA

6.2.1. Calibration Standards

6.2.1.1. Instrument calibration is necessary to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet contract required quantitation limits (CRQLs). Calibration establishes the dynamic range of an

instrument, establishes response factors to be used for quantitation, and demonstrates instrument sensitivity. Criteria for calibration are specific to the instrument and the analytical method. Each instrument shall be calibrated according to the manufacturer's guidelines using standard solutions appropriate to the type of instrument and the linear range established for the analytical method. Calibration procedures for the methods included in this QAPP are included in the corrective action summary tables in Appendices A through H. The calibration acceptance criteria and corrective actions for instrument calibration are also included in Appendices A through H. The following paragraphs describe the general requirements for standards preparation and traceability and laboratory instrument calibration procedures.

6.2.1.2. Data accuracy is dependent upon the accuracy of the standards used for instrument calibration. To ensure the highest quality standard, primary reference standards used by the Contract Laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA Cooperative Research and Development Agreement (CRADA) vendors, American Association of Laboratory Accreditation (AALA) vendors, or other reliable commercial sources. When standards are received at the laboratory, the date received, supplier, lot number, purity, concentration, and expiration date shall be recorded in a standards log book. Vendor certifications for the standards shall be retained in the files.

6.2.1.3. Standards shall be obtained in their pure form or in a stock or working standard solution. All records regarding standards shall unambiguously trace their preparation, use in calibration, expiration dates, and quantitation of sample results. All standards shall be given a standard identification number and the following information recorded in the appropriate file (standards logbook): source of standard, the initial concentration of the standard, the final concentration of the standard, the volume of the standard that was diluted, the solvent and the source and lot number of the solvent used for standard preparation, the expiration date of the standard, and the preparer's initials.

6.2.1.4. After preparation and before routine use, the identity and concentration of the standards shall be verified. Verification procedures include a check for chromatographic purity (if applicable) and verification of the standard's concentration by comparing its response to a standard of the same analyte prepared or obtained from a different source. Reagent purity shall be assessed by analyzing an aliquot of the reagent lot using the analytical method in which it shall be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory. Standards shall be routinely checked for signs of deterioration (e.g., discoloration, formation of precipitates, and changes in concentration), and shall be discarded if deterioration is suspected or the expiration date has passed. Expiration dates may be taken from the vendor recommendation, the analytical methods, or from internal research.

6.2.2. Instrument Calibration

6.2.2.1. Instrument calibration is necessary to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet CRQLs. Calibration establishes the dynamic range of an instrument, establishes response factors to be used for quantitation, and demonstrates instrument sensitivity. Each instrument shall be calibrated according to the manufacturer's guidelines using standard solutions appropriate to the type of instrument and the linear range established for the analytical method. The instrument calibration shall be from the lowest to the highest calibration standard and the lowest calibration standard concentration shall be at or below the CRQL for each target analyte.

6.2.2.2. All instrument calibration information shall be documented, and at a minimum include the equipment to be calibrated, the reference standards used for calibration, the calibration techniques, actions, acceptable performance tolerances, frequency of calibration, and calibration documentation format. The laboratory shall

maintain records of standard preparation and instrument calibration. Calibration records shall include daily checks using standards prepared independently of the calibration standards, and instrument response shall be evaluated against established criteria. The analysis logbook, maintained for each analytical instrument, will include at a minimum the date and time of calibration, the initials of the person performing the calibration, and the calibrator reference number and concentration. Calibration procedures for the methods included in this QAPP are presented in Appendices A through H and are from the following sources:

- EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), (U.S. EPA Third edition, September 1986a; Final Update I, July 1992; Final Update IIA, August 1993; Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996)
- EPA 100-400 Series - Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600R-93/100, August 1993a)
- EPA 200 Series - Methods for the Determination of Metals in Environmental Samples, (EPA/600/4-91-010, June 1991a; Supplement I, EPA/600/R-94/111, May 1994)
- EPA 600 Series - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (U.S. EPA, CFR Title 40, Part 136, Appendix A, July 1996)
- Compendium of Methods for Determination of Toxic Organic Compounds in Ambient Air (EPA/600/4-89/017, June 1988b)

- State of California, Department of Health Services, Determination of Perchlorate by Ion Chromatography (Rev. No. 0, June 1997).

The QC acceptance criteria and corrective actions for instrument calibration are included in Appendices A through H.

6.2.2.3. Frequency of instrument calibration and QC sample analysis for the analytical methods are batch controlled. All definitive data for Hill AFB projects shall be associated with sample batch QC samples that were extracted concurrently with the site samples and analyzed sequentially in the same analytical batch, and on the same instrument as the primary samples. The following paragraphs define sample and instrument batches.

6.2.2.4. For Hill AFB projects, a sample batch is a group of 20 or fewer samples of the same matrix which are extracted concurrently, or in limited continuous sequential time periods. Keeping batches “open” for more than two hours shall not be acceptable; samples and their associated QC samples (method blank, LCS, and MS/MSD) shall be prepared in a continuous process. The sample batch shall be analyzed sequentially on a single instrument as practicable.

6.2.2.5. For Hill AFB projects, the instrument batch shall be defined as a group of 20 or fewer samples which are analyzed together within the same analytical run sequence as defined by the method calibration criteria, or in a sequential time period. Samples in each batch are of similar matrix (e.g., soil, sludge, liquid waste, water), are treated in a similar manner, and the same reagents are used.