U.S. Army Environmental Center

U.S. Army Environmental Center Guidelines for Implementation of ER 1110-1-263 for USAEC Projects

May 1993

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Prepared for
U.S. Army Environmental Center
Aberdeen Proving Ground, MD 21010-5401

AEC Form 45, 1 Feb 93 replaces THAMA Form 45 which is obsolete.

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FOREWORD

This guidance document describes how ER 1110-1-263, Chemical Data Quality Management For Hazardous Waste Remedial Activities, shall be implemented for projects being performed for the U.S. Army Environmental Center (USAEC) Installation Restoration and Base Closure Projects. The Quality Assurance Project Plan submitted in fulfillment of a project requirement should be a detailed, step-by-step document implementing the procedures described herein.

The primary purpose of this document is to comply with EPA requirements. In addition, the concepts expressed in this document represent what is considered by the USAEC to be the best general approach for implementing the requirements of ER 1110-1-263.

Modifications to the requirements in this document may be made to meet program and/or project specific requirements, such as those specified by the EPA Region, or state authority. All modifications must be co-ordinated with, and approved by the USAEC Chemistry Branch through the Contracting Officer's Representative (COR)/project officer.

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NOTE ON SUBMISSIONS TO USAEC

NOTE: Whenever submission of material is required for USAEC review, decision, or approval; the contractor shall submit two copies, one to the Contracting Officer's Representative (COR) and one to Chemistry branch. In certain cases the material to be reviewed may be supplied to only one party, however, the cover letter must be supplied to both parties. The exact procedures to be followed will be determined for each project. Chemistry Branch will forward their replies through the COR/project officer. Responses are not official unless signed by the COR.

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2.0 QUALITY ASSURANCE PROJECT PLAN

2.1 INTRODUCTION

Prior to initiating field sampling and analysis of environmental samples, the Contractor Laboratory shall develop a detailed Quality Assurance Project Plan (QAPjP) for the specific project being supported. The QAPjP will be submitted to the USAEC Project Officer or the Contracting Officer's Representative, who will forward the plans to the Chemistry Branch for approval. Although ER 1110-1-263 and these guidelines outline a system for verifying and maintaining a desired level of performance quality, the QAPjP must provide laboratory-specific descriptions of how these will be implemented.

2.2 PURPOSE

The purposes of the Quality Assurance Project Plan are to:

- Be compatible with EPA and/or state requirements.
- Establish function-specific responsibilities and authorities for data quality;
- Establish procedures to ensure that all data are collected under conditions of analytical system control;
- Establish procedures for recognizing and correcting out-of-control situations;
- Establish procedures to ensure that non-laboratory activities do not compromise analytical data quality; and
- Establish record keeping procedures commensurate with project data uses.

2.3 CONTENTS

The USAEC Chemistry Branch recognizes that implementation of these guidelines will vary between laboratories. The structure of the QA/QC organization will depend not only on laboratory differences, but also on the contractor's project structure. For these reasons, the QAPjP must address laboratory-specific and project-specific situations that are not addressed by these guidelines.

The QAPjP shall include, as a minimum, the following information and descriptions in accordance with EPA QAMS 005/80:

- Title page with provision for approval signatures;
- Table of contents;
- Project description;
- Project organization and responsibility;
- QA objectives for the measurement of data in terms of precision, accuracy, completeness, representativeness, and comparability;
- Sampling procedures;
- Sample custody;
- Calibration procedures and frequency;
- Analytical procedures;
- Data reduction, validation, and reporting;
- Internal quality control checks and frequency;
- Performance and system audits and frequency;
- Preventive maintenance procedures and schedules;

- Specific routine procedures to be used to assess data precision, accuracy, and completeness of specific measurement parameters involved;

- Corrective action; and

- Quality assurance reports to management.

In addition the following information and descriptions should be included:

- A statement of adherence to or reference to ER 1110-1-263 and these guidelines;

- A detailed account of how the contractor, in conjunction with any subcontractors, will implement these guidelines;

- A description of sampling team and analyst training in technical skills, standard QC, and essential elements of theses guidelines;

- QC sample introductions and lot sizing;

- A description of applicable logs (field, instrument, sample, QC) and their use;

- Storage and use of standard analytical reference materials;

- A list of personnel responsible for data review and sequence of review prior to submittal; and

- A list of SOPs.

Not all of these items are addressed in this document, but are part of good laboratory practices and must be included in the QAPjP. Whenever possible and appropriate, names of individuals and step-by-step procedures should be provided. Any changes to an approved QAPjP must be requested in writing, approved by the USAEC Chemistry Branch, and formally coordinated through the Project Officer or Contracting Officer's Representative (COR). Written approval from USAEC must be obtained prior to implementation of the requested change. In the event that timely implementation is essential, verbal approvals may be granted on a limited basis provided the changes do not impact on resources or costs. These informal requests for changes and approvals will be formalized immediately in writing in order to document the change.

3.0 SAMPLE COLLECTION AND MANAGEMENT

3.1 INTRODUCTION

The procedures described in this section are designed so that the samples obtained will be proper representations of the matrix being sampled. Trace levels of contaminants from sources external to the sample must be eliminated through the use of good sampling techniques. Sample management and stringent documentation are the key factors in a successful QA program for sampling.

This section does not discuss sampling of air or biological matrices, or sampling for radiological constituents. When these matrices or analytes are included in a project, detailed requirements and protocols will be provided on a case-by-case basis. References are provided in the Bibliography which should be consulted when planning air or biological sampling (ASTM, 1973; EPA, 1974; EPA, 1976; EPA, 1977b; EPA, 1977c; EPA, 1978; EPA, 1983c; EPA, 1983d; U.S. Geological Survey, 1977a; and Weber, 1972).

Sampling requirements vary according to the analytes of interest and the environmental matrices sampled. These differences are discussed in Section 3.4 to 3.9. Section 3.3 discusses sample containers and Section 3.10 discusses sample preservation. References are provided in the Bibliography that discuss appropriate sampling methods in detail. These references should be consulted when preparing sampling plans (Barcelona et al., 1984; Nielson and Yeates, 1985; EPA, 1977a; EPA, 1980c; EPA, 1982a; EPA, 1982b; EPA, 1982d; EPA, 1983b; EPA, 1984a; EPA, 1984c; and U.S. Geological Survey, 1977b). The specific procedures which will be used must be described in detail in the Sampling and Analysis Plan (SAP), and the QAPjP.

NOTE: Due to variances in sample collection protocol among EPA regions and State agencies, the following collection procedures are provided as default parameters. Variances necessary to meet Regional or State requirements should be considered and identified for USAEC review and approval.

Documentation of sampling activities is described in Section 3.13.

3.2 PERSONNEL

It is the responsibility of the contractor to establish personnel qualifications and training requirements for all positions. Each member of the field team shall have the education, training, technical knowledge, and experience, or a combination thereof, to enable that individual to perform assigned functions. Personnel qualifications shall be documented in the sampling plan in terms of education, experience, and training. Training shall be provided for each team member as necessary to properly perform their functions. The suggested minimum qualifications are as follows:

- Geologist - Baccalaureate Degree in Geology, Geotechnical Engineering, or Geohydrology.

- Sampler 3 - High School Degree or equivalent plus 40 hours of OSHA training plus at least 16-hours instruction in sample collection techniques.

- Sampler 2 - All requirements for Sampler 3 plus 6-months experience (minimum participation in 3 sampling events) as Sampler 3.

- Sampler 1 (Team Leader) - All requirements of Sampler 2 plus 4-hour class in chain-of-custody procedures plus an additional 6-months experience (minimum participation in six sampling events) as Sampler 2. A Baccalaureate Degree in an Engineering or Science related subject is desirable.

3.3 CONTAINERS

Sample containers shall be chosen in accordance with Appendix F of ER 1110-1-263 and must be compatible with EPA requirements. However, for all USAEC projects 3 separate 40 ml vials shall be used for the collection of all water samples for volatile analysis.

All sample containers shall be cleaned before use according to the protocols specified by the EPA's Contract Laboratory Program (see Appendix C and S).

3.4 VOLATILES

The field sampling checklist (Appendix S) should be used to verify that all sampling is performed correctly.

3.4.1 GROUND/SURFACE WATER SAMPLES

When sampling water for volatile compounds, extra care must be exercised to prevent analyte loss by evaporation or by agitation of the sample. Precautionary measures include:

- Acquiring the sample with equipment that minimizes water gas/liquid interphase under pressure or vacuum;

- Avoiding aeration or agitation of the sample to the greatest possible extent;

- Taking triplicate samples, as a minimum;

- Filling vials to capacity, taking care that no air bubbles are trapped in the vial;

- Preserving to pH 2 or less with sodium bisulfate or HCl (NOTE - this procedure is not to be used for any sample from an area of suspected agent Mustard (HD) contamination site or any site potentially containing the Mustard breakdown product thiodiglycol. This will reduce the holding time to 7 days);

- Turning vial over and tapping gently against a hard surface or hand. If air bubbles are trapped in the vial, discard and take another sample. Repeat until triplicate samples, free of air bubbles, are obtained;

- As each vial is correctly filled, entering the applicable information on the label and then packing the vial into the shipping container. The contents of the shipping container must be kept at the required temperature at all times.

- Storing the sample at 4°C;

• Analyzing the sample as soon as possible, and never exceeding the prescribed holding time (Section 6.5);

• Never allowing a volatile sample to freeze (this includes **any** ice formation in the sample bottle); and

• Never filtering the sample.

3.4.2 TAP WATER SAMPLES

The following procedures are to be used in the sampling of water from taps located anywhere in a water supply system:

• Water should be allowed to run from the tap for 2 to 3 minutes before sampling;

• Remove the aerator from the tap, if possible;

• Slow the water flow to a trickle before filling the sample vial;

• Fill vial to the top, forming a water bulge above the rim. Add sodium thiosulfate to stop the chlorine reaction, as required. Screw on the cap without dislodging the teflon liner;

• Turn vial over and tap gently against a hard surface or hand. If air bubbles are trapped in the vial, discard and take another sample. Repeat until triplicate samples, free of air bubbles, are obtained; and

• As each vial is correctly filled, enter the applicable information on the label and then pack the vial into the shipping container. The contents of the shipping container must be kept at the required temperature at all times.

3.4.3 SOIL AND SEDIMENT SAMPLES

The sampling method for volatiles in soil or sediment will depend on the chemical analysis procedure and the nature of the soil or sediment. Portions of soil may be placed in empty vials containing the extraction solvent. In other instances, sealed cores may be shipped to the laboratory for subsampling. Acceptable materials for sealing cores must be approved by USAEC and interested regulatory agencies on a project specific basis.

The primary considerations for acquiring samples for volatiles, either in the field or in the laboratory, include the following:

- Samples stored at 4°C;
- Sample handling should be minimized;
- Sample/air contact should be minimized;
- The sample or subsample should be placed in an air-tight container immediately after collection.
- Air-tight seals on all containers used in shipment or laboratory workup.

3.5 GROUNDWATER

All groundwater sampling will occur after the wells have been developed according to the USAEC Geotechnical requirements document and/or specifications in the contract. Because drilling and well construction disturb the natural groundwater system, the maximum possible length of time (never less than two weeks, unless an waiver is obtained from the COR) shall pass between well development and sampling to allow the groundwater system to return to chemical equilibrium. The field sampling checklist (Appendix S) should be used to verify that all sampling is performed correctly.

3.5.1 MONITOR WELLS

The following procedures incorporate the necessary aspects of sampling QA and shall be used each time a monitor well is sampled:

- Measure the depth from the top of the well casing (not protective casing) to the top of the water and record the depth in the sampling logbook;

- Measure and record the depth from the top of the casing to the bottom of the sediment/water interface;

- Subtract the depth to top of the water from the depth to the bottom of the sediment/water interface to determine the height of standing water in the casing and saturated annulus. Remember to have on hand the diameter, height, and porosity of the sand pack, as recorded by the geologists during well construction;

- Obtain a sample of groundwater for temperature, conductivity, and pH measurements. Record these measurements in the sampling logbook;

- Remove a quantity of water from the well equal to 5 times the calculated volume of water in the well, including the saturated annulus;

- If the well goes dry during pumping or bailing, one is assured of removing all water which had prolonged contact with the well casing or air. If the recovery rate is rapid, allow the well to recover to its original level and purge a second time before sampling. If recovery is very slow, samples may be obtained as soon as sufficient water is available;

- Obtain samples for chemical analysis immediately after pumping or bailing is complete. For slow recovering wells, the sample shall be collected immediately after a sufficient volume is available;

- After obtaining chemical analysis samples, draw a second sample for temperature, conductivity, and pH measurement and record results in the sampling logbook;

- Filter samples, as appropriate; samples to be analyzed for VOCs should never be filtered;

• All samples must be placed in containers as specified in Appendix F of ER 1110-1-263. Except for volatiles, the sample bottle and cap shall be triple rinsed with the water being sampled before filling the bottle with the sample to be analyzed. Sample container for volatiles are never rinsed. Bottles for filtered samples shall be rinsed with filtered sample water and bottles for unfiltered samples shall be rinsed with unfiltered sample water (these requirements may be waived if it is not permitted by the regulatory agency having jurisdiction);

• Add the appropriate preservative and cap securely;

• Label samples in accordance with Section 3.13; and

• Place sample bottle(s) in a temperature controlled (4°C) chest immediately after sampling and deliver to the laboratory as soon as possible, in accordance with the chain-of-custody requirements specified in section 4.0 and Appendix E.

Note that the rinsing requirement specifically precludes adding preservative to bottles before they are shipped to the sampling site. The sampling team must have available the correct preservatives and must be trained in handling and dispensing the preservatives (Field Sampling Checklist, Appendix S).

3.5.2 WATER SUPPLY WELLS

The following procedures incorporate the necessary aspects of sampling QA and shall be used each time a water supply well is sampled:

• From existing well data or an estimated well depth, calculate the maximum possible volume of water in the well casing;

• Obtain a sample of groundwater for temperature, conductivity, and pH measurements. Record these measurements in the sampling logbook; and

• Pump to discard at least 5 times the estimated volume of water in the well.

• Filter samples, as appropriate; samples to be analyzed for VOCs should never be filtered;

• All samples must be placed in containers as specified in Appendix F of ER 1110-1-263. Except for volatile samples the sample bottle and cap shall be triple rinsed with the water being sampled before filling the bottle with the sample to be analyzed. Sample containers for volatiles are never rinsed. Bottles for filtered samples shall be rinsed with filtered sample water and bottles for unfiltered samples shall be rinsed with unfiltered sample water (these requirements may be waived if it is not permitted by the regulatory agency having jurisdiction);

• Add the appropriate preservative and cap securely;

• Label samples in accordance with Section 3.13;

• Place sample bottle(s) in a temperature controlled (4°C) chest immediately after sampling and deliver to the laboratory as soon as possible, in accordance with the chain-of-custody procedures specified in section 4.0; and

• After obtaining chemical analysis samples, draw a second sample for temperature, conductivity, and pH measurements and record results in the sampling logbook.

Note that the rinsing requirement specifically precludes adding preservative to bottles before they are shipped to the sampling site. The sampling team must have available the correct preservatives and must be trained in handling and dispensing the preservatives.

Prior to taking samples, ensure that the water to be sampled is raw (untreated) water. Under no circumstances should treated water be taken for chemical analysis to define the levels of contamination in the aquifer. If holding or pressure tanks are used in the water supply system, they should be bypassed to obtain good representative groundwater samples.

3.5.3 TAP WATER

The following procedures are to be used in the sampling of water from taps located anywhere in a water supply system:

- Water should be allowed to run from the tap for 2 to 3 minutes before sampling;

- Except for volatile samples, triple rinse sample vial with sample water (this requirement may be waived if it is not permitted by the regulatory agency having jurisdiction). Sample containers for volatiles are never rinsed;

- Each sample container must be completely filled with the water sample;

- Conductivity, pH, and temperature measurements, if required, must be performed on the water samples collected for inorganic analysis; and

- As each vial is filled, enter the applicable information on the label and then pack the vial into the shipping container. The contents of the shipping container must be kept at the required temperature (4°C) at all times.

- Ship all samples to the laboratory in accordance with the chain-of-custody procedures specified in section 4.0.

Note that the rinsing requirement specifically precludes adding preservative to bottles before they are shipped to the sampling site. The sampling team must have available the correct preservatives and must be trained in handling and dispensing the preservatives. If drinking water quality is to be determined, the sampled tap(s) must be located after any water treatment processes.

3.6 SURFACE WATER

Surface water samples may be obtained under many different circumstances. At the time of sampling, the procedures described in the Project QC Plan and Project Workplans shall be followed. These documents must have designated the appropriate techniques for the project-specific setting, as described in Section 3.1. The field sampling checklist (Appendix S) should be used to verify that all sampling is performed correctly.

Before sampling, equipment shall be rinsed downflow or away from the sampling point, taking care not to disturb sediments at the sampling point. After sampling at each location, the equipment shall be rinsed with distilled water or USAEC-approved water, as discussed in Section 3.11.

All samples shall be placed in the appropriate containers as specified in Appendix F of ER 1110-1-263. The need for sample filtration will be determined according to the requirements given in Section 6.8 or as specified in the task order. Except for volatile samples, the sample bottle and cap shall be triple rinsed with the water being sampled before filling the bottle with the sample to be analyzed. Sample containers for volatiles are never rinsed. Bottles for filtered samples shall be rinsed with filtered sample water and bottles for unfiltered samples shall be rinsed with unfiltered sample water (this requirement may be waived if it is not permitted by the regulatory agency having jurisdiction). Add the appropriate preservative and cap securely. Samples must be labeled in accordance with Section 3.13. The sample bottle(s) shall be placed in a temperature controlled (4°C) chest immediately after sampling and delivered to the laboratory as soon as possible, in accordance with the chain-of-custody procedures specified in section 4.0 and Appendix E.

Note that the rinsing requirement specifically precludes adding preservative to bottles before they are shipped to the sampling site. The sampling team must have available the correct preservatives and must be trained in handling and dispensing the preservatives (Field Sampling Checklist, Appendix S).

3.7 SOILS

The sampling team is responsible for collecting representative samples that can be analyzed as received from the field. The Program Manager, Sampling Team Leader, and Contractor QAC must train the sampling team in the types of soils to be collected, the components of interest in the samples, and how to collect the sample that will represent the matrix of interest. Specifically, the sampling team must be trained to remove all items that are not integral components of the matrix of interest.

The Quality Assurance Project Plan and Workplans must have considered appropriate sampling distributions and techniques, as described in Section 3.1. The sampling locations must have been chosen to be representative of the areas being investigated. At the time of sampling, these plans shall be followed. A large area may require collecting and compositing multiple samples into a single sample to represent the area. Individual samples may be collected and analyzed to describe the sampling points within the area. The field sampling checklist (Appendix S) should be used to verify that all sampling is performed correctly.

All sampling points must be marked with a stake that is labeled with the appropriate Site Identification. Prior to sampling, surface vegetation, rocks, pebbles, leaves, twigs, and debris will be cleared from the sample point to allow collection of a representative soil sample. After sampling each location, all equipment must be thoroughly cleaned to prevent cross-contamination of samples. Equipment shall be scrubbed and rinsed with distilled water or USAEC approved water, as described in Section 3.11.

Soil samples taken from borings shall be obtained via a split or solid barrel sampler (e.g., Split-Spoon, Dennison, Pitcher), or sampler equipped with a polybutyrate (or similar) liner. Borings shall be produced in a manner that preserves sample integrity and composition. Upon reaching the surface, the sampler shall be opened and the sample extracted, peeled, and bottled in the shortest possible time and placed in a cooler at 4°C. In the case of the polybutyrate liner, ends shall be capped and taped and placed in a cooler at 4°C. Detailed instruction on the handling of samples using these liners shall be provided in the project sampling plan if their use is required. Peeling is the process that removes the portion of sample which is in direct contact with the sampler. In addition, the ends of the sample are removed and discarded. Samples for volatiles analysis shall be peeled, bottled, and capped within 15 seconds from the time the sampler is opened.

Soil samples shall be placed in appropriate containers as specified in Appendix F of ER 1110-1-263. Samples for volatile organics shall be placed in containers appropriate for the analytical method (Section 3.4.3). Samples must be labeled in accordance with Section 3.13. Sample bottles shall be placed in a temperature controlled (4°C) chest immediately after sampling and delivered to the laboratory as soon as possible, in accordance with the chain-of-custody requirement specified in section 4.0 and Appendix E.

3.8 SEDIMENTS

The sampling team is responsible for collecting representative samples that can be analyzed as received from the field. The Program Manager, Sampling Team Leader, and Contractor QAC must instruct the sampling team in the types of sediments to be collected, the components of interest in the sample, and how to collect the sample that will represent the matrix of interest. Specifically, the sampling team must be trained to remove all items that are not integral components of the matrix of interest.

The type of sampler to be used will be dictated by the nature, as well as the accessibility, of the sediments. In addition, the type of sampler chosen should be appropriate for obtaining the desired sample, e.g., a core sampler should not be used to obtain top sediment. The Project QC Plan and Workplans should have designated appropriate sampling techniques, as described in Section 3.1. At the time of sampling, these plans must be followed. The field sampling checklist (Appendix S) should be used to verify that all sampling is performed correctly.

Prior to sampling sediments in a stream, the sampling device shall be rinsed with stream water at a point downstream from the sampling location to avoid disturbing the sediments at the sampling point. Also, sampling shall be accomplished upstream of any disturbances in the stream caused by the sampler or sampling team. Twigs, leaves, pebbles, and debris that are not integral components of the matrix of interest must be removed by the sampling team.

Prior to sampling sediments in a pond or lagoon, the sampling device shall be rinsed with water near the sampling point. However, caution must be exercised to avoid disturbing the sediments at the sampling point by the rinsing activities.

After sampling each location, all equipment must be thoroughly cleaned to prevent cross contamination of samples. Equipment shall be scrubbed and rinsed with distilled water or USAEC-approved water, as described in Section 3.11.

Sediment samples shall be collected in appropriate containers as specified in Appendix F of ER 1110-1-263. Samples must be labeled in accordance with Section 3.13. Sample bottles shall be placed in a temperature controlled (4°C) chest immediately after sampling and delivered to the laboratory as soon as possible.

3.9 SURFACE WIPE SAMPLES

Surface wipe samples shall be collected in accordance with the following guidelines:

- Wiping media (ie. filter paper, cotton balls, or gauze pads) shall be chosen to be compatible with the surface(s) being wiped. A sample(s) of the media shall be submitted each day as a media blank(s). Media blank(s) shall be analyzed for all analytes of interest sampled on that day;

- An appropriate wiping solvent shall be chosen for each class of sample to be collected. The choice of solvents shall be specified in the QAPjP (in general, a 1:4 acetone/hexane mixture should be used to wipe for organic analyses and deionized water should be used to wipe for inorganics). A solvent blank shall be submitted for each lot of solvent used and shall be analyzed for all project analytes of interest;

- Templates should be used to ensure that the area wiped is consistent from site to site. The suggested standard area (based upon industrial hygiene standard practice) is 100 square centimeters;

- Wiping should be done in a systematic fashion. The area should first be wiped horizontally from top to bottom, then vertically from left to right. After wiping is completed, the wipe shall be placed in an appropriate sample container and placed in a cooler at 4°C. No other preservation is required;

- The wiping media may be handled either with tongs or held in a gloved hand. If the media is held directly in a gloved hand then a "glove blank" shall be submitted for

analysis with each day's samples. This shall consist of wiping solvent poured over a clean pair of gloves and collected in the appropriate container. If more than one solvent is in use, a blank shall be collected for each solvent.

3.10 SAMPLE PRESERVATION

The purpose of sample preservation is to prevent or retard the degradation/modification of chemicals in samples during transit and storage prior to analysis. Efforts to preserve the integrity of the samples shall be initiated at the time of sampling and will continue until analyses are performed. Preservatives shall be added to the sample container at the time of sample collection. The recommended procedure for accomplishing this is to take premeasured volumes of the preservatives in sealed ampules to the field. Preservation and storage requirements are provided in Appendix F of ER 1110-1-263. Sample holding time requirements, as listed in Section 6.5, apply to all samples. Holding times begin on the sampling date and not the date samples are received in the laboratory. Freezing samples to extend holding times shall not be permitted.

Note that samples for volatiles and TOC which are collected from areas of suspected agent Mustard (HD) or thiodyglycol contamination are not to be preserved, due to the possibility of Mustard reformation in the presence of hydrochloric acid.

Sample storage shall only be terminated after all analytical results have been validated to level 3 in the USAEC Data Management System and approved by the USAEC Project Officer. Samples may be required to be held in storage longer to fulfill contractual requirements or as directed by the USAEC Project Officer/COR.

3.11 EQUIPMENT DECONTAMINATION

All equipment used to measure and sample the groundwater system (e.g., bailers, pumps, tapes, ropes) must be cleaned before use in each well to prevent cross contamination between wells. Equipment that is dedicated to a well site may not require cleaning between sampling events. If the well is free of inflowing sediments, thorough rinsing will be sufficient. When inflowing sediments adhere to equipment, scrubbing may be required in addition to rinsing. In no instance shall detergents, soaps, or solvents be used to routinely clean equipment in the field, without approval of USAEC Chemistry Branch through the COR/project officer. At sites where known cleaning problems exist the use of extra cleaning agents may be proposed in the QAPjP.

Water used for rinsing field equipment shall be bottled distilled water or water from a USAEC-approved source. Such USAEC-approved water should originate from an uncontaminated (background) and untreated (unchlorinated) source. The water shall be analyzed by a Missouri River Division (MRD) validated laboratory for all project specific analytes prior to collection of field samples. Water from chemical supply companies or retail merchants is acceptable, provided that analysis by an MRD validated laboratory reveals such water is free of interferences. At least one sample must be submitted to the laboratory and be analyzed for all analytes of interest prior to the first use in the field. The initial rinse water analyses may be done prior to completion of laboratory validation, provided that the analytical procedures used are identical to those to be validated. A rinse water sample shall be included with the first lot of samples during the initial and subsequent sampling excursions, defined as the time between mobilization and demobilization of the sampling team. Additional rinse water samples shall be taken, as required, to meet the DQOs of the project. Waivers to these requirements will be considered by the USAEC Chemistry Branch through the COR/project officer on a case-by-case basis.

Sampling equipment must be protected from ground surface contamination. Clean plastic sheeting spread around the well is one means of protecting the equipment. New protective sheeting should be used at each sampling location. Sampling efforts shall preclude wind-blown particles from contaminating the sample or sampling equipment.

Exceptions to this policy shall only be implemented after receipt of written approval from the Chemistry Branch of USAEC through the COR/project officer.

3.12 STANDING OPERATING PROCEDURES - FIELD

The contractor shall have written SOPs for all field procedures and methods; all procedures shall be performed as described in the SOP. Any modification of an SOP made during a data collection activity must be documented and approved by the USAEC Chemistry Branch through the COR. SOPs shall be prepared for, but not be limited to, the following areas:

- Sample management;
- Sample team training and documentation;
- Numbering and labelling of samples;
- Sample tracking;
- Sample containers;
- Sample preservation and storage;
- Holding times;
- Shipping;
- Decontamination;
- Sample collection procedures;
- Corrective actions;
- Records management;
- Chemical and sample disposal; and

- Reporting.

In addition, where analyses are performed in the field, the following additional SOPs are required:

- Reagent/standard preparation and validation;
- Equipment calibration and maintenance;
- Field analysis; and
- Data reduction and validation.

A description of the basic information required in each of the above SOPs is included in Appendix D. The contractor's SOP is not required to conform to a specific format but shall be representative of good standard field and laboratory operations, and shall give clear evidence of the contractor's ability to successfully fulfill all contract requirements.

3.13 SAMPLE MANAGEMENT

3.13.1 FIELD CHAIN-OF-CUSTODY

The necessity of having established procedures for documenting activities in the field also requires that each sample taken be delivered to the laboratory. To alleviate potential problems, the field sampling team must adequately document and identify each sample taken. This process ensures that each sample is analyzed for the requested parameters by the laboratory, and each sample requested is actually received at the laboratory. It is imperative that written procedures be not only available but followed, to ensure that an accurate record of sample collection and transfer activity is maintained. Chain-of-custody procedures are contained in Section 4.0 and Appendix E.
All required information listed in Section 4.0 shall be included on all chain-of-custody forms.

3.13.2 SAMPLE HANDLING

It is important to good custody procedures that all samples be handled by a minimum number of persons. Field records must be completed at the time a sample is collected and should include the following information as a minimum:

- Project or installation for which the sample is being taken;
- Sample date and time;
- Sample location (bore or well i.d.) or source;
- Field sample number, unique to each sample location;
- Required analyses for each container;
- Preservative used, if any;
- Field data applicable to the sample (i.e., pH, conductivity); and
- Sampler's name (the individual who actually fills the sample container).

Additional information which is required for certain samples such as wells or bore holes, would include:

- Sample depth, measured from the top of the well casing for established wells, and from ground level for bores; and
- Sample technique.

Information which is entered on the field chain of custody must match exactly the information from the field sampling log. All entries must be made in blue or black ink, and must be legible. There shall be sufficient matching information on each sample label to verify each sample against the chain of custody. As a minimum, the following information is required:

- Sample date and time;

- Sample location (bore or well i.d.) or source;

- Field sample number, unique to each container, if several analytical samples are being taken from the same source;

- Required analyses for each container;

- Preservative used, if any; and

- Sampler's name or initials.

Unused bottles, containers, and coolers which have been shipped to a sampling location are to be kept in a secured location to minimize tampering and possible contamination.

When samples are to be transferred, the custodian must sign and date the chain-of-custody form(s), as must the recipient who now becomes the sample custodian. Transfers must account for each individual sample, even when samples are transferred as a group.

Shipped packages are considered under chain-of-custody if the carrier signs a form indicative of receipt; a receipt is also generated by delivery of the samples. This receipt is attached to the original chain-of-custody forms, which shall be shipped inside of the cooler or container to prevent loss upon transfer. Custody seals should be placed across all edges of the cooler lid except for the hinge side, to ensure that no tampering has occurred.

3.13.3 SAMPLE RECEIPT

When samples are received at the analytical laboratory the coolers shall be inspected as soon as possible and the following information recorded:

- Condition of cooler, including whether custody seals are intact.

- Whether chain-of-custody documents are enclosed in the cooler and are properly filled out.

- Whether sample containers are intact and sealed with evidence tape.

- Temperature of cooler. This should be measured in a separate container (temperature blank) and not in an actual sample.

The USAEC requires that all samples be cooled to 4° C. However, samples received at up to 6° C may be analyzed. Any samples received at temperatures greater than 6° C shall not be analyzed without the approval of the USAEC project officer/COR and the USAEC Chemistry Branch. Any sample received which exhibits any signs of icing shall not be analyzed without the approval of the USAEC project officer/COR and the USAEC Chemistry Branch.

All sample receipt information shall be recorded on an appropriate form and placed in the lot data package. Whenever discrepancies are found a non-conformance report to management shall be generated, a copy of which shall be maintained in the lot data package.

4.0 CHAIN-OF-CUSTODY PROCEDURES

All work performed for the USAEC shall adhere to the chain-of-custody procedures specified in NEIC Policies and Procedures (EPA-300/9-78-001-R). See Appendix E for a summary of these requirements.

At a minimum, the following information shall be recorded on the chain-of-custody form:

- Date of Sampling
- Matrix type (3 characters)
- Site type (4 characters)
- Site Identification Number (10 characters)
- Depth (in the format XXX.X)
- Sample Technique (1 character)
- Analysis Required (should specify specific method)
- Installation (2 characters)
- Prime Contractor (2 characters)
- Sampling Program (3 characters)
- Field Sampling Number (Optional)

Figure 4-1 illustrates a chain-of-custody form which meets the above requirements.

Figure 4-1

ZYX Engineers
CHAIN OF CUSTODY RECORD - USAEC SAMPLES

Installation (2):
Sample Program (3):
Laboratory (2):

COC By: ____

ZYX Field Sampler Initials (3)	Time of Sampling (Military Clock) (4)	Site ID (10)	ZYX Field Sample ID (8)	File Type (3)	Site Type (4)	Depth Ft. (5)	Sample Techniques (1) / Sample Flag Code (1) / Sample QC Code (1) / TEST NAME / CODE																		Matrix	Number of Containers

Relinquished by: ZYX Engineers

Date: _____ Time: _____ Signature: _____

Accepted by:

Date: _____ Time: _____ Signature: _____

Accepted by:

Date: _____ Time: _____ Signature: _____

Carrier: _____ Air Bill Number: _____

Total No. of Containers

Relinquished by:

Date: _____ Time: _____ Signature: _____

Comments:

5.0 LABORATORY VALIDATION

5.1 INTRODUCTION

Before using an analytical method to analyze environmental samples, a Contractor Laboratory must demonstrate the ability to perform the method for specific analytes, and, in the process, generate data to be used in establishing Method Detection Levels (MDLs). Standardized analytical methods shall be selected from the EPA's Contract Laboratory Program (CLP), SW-846, or from some other EPA standard method (ie. 200, 500, and 600 series). If the analyte of interest is not addressed in either of the above sources, then methodology will be provided by the USAEC, if available. The USAEC will also provide a list of the target analytes for all USAEC work and Required Detection Levels (RDLs). RDLs are defined as the lowest level required by any federal or state regulations, that is technically achievable with available instrumentation.

Laboratory validation is a three phase process involving an initial validation of the laboratory by the U.S. Army Corps of Engineers Missouri River Division (MRD), the determination of method detection levels(MDLs), and the documentation of methods to the USAEC. The laboratory shall demonstrate its ability to perform the analysis for specified compounds using the standardized methods. A normal timeframe for completion of this process is 12 to 18 weeks.

Due to the constraints of sample holding times as specified in ER 1110-1-263, collection of environmental samples shall never occur before all required analytical methods are validated.

5.2 VALIDATION PROCEDURES

5.2.1 MRD LABORATORY VALIDATION

MRD validation procedures are described in detail in Appendix C of ER 1110-1-263. To summarize, this is a 3 step process;

- The laboratory must submit its Quality Management or Quality Assurance Manual to MRD for review;

- MRD will provide the laboratory with performance audit (PA) samples, which the laboratory shall analyze according to the method specified by MRD (NOTE: This is not necessarily the method selected by the laboratory for routine use);

- Upon successful analysis of the PA samples, a representative of MRD will visit the laboratory for an on-site inspection.

5.2.2 METHOD DETECTION LEVEL

The laboratory shall determine a Method Detection Level (MDL) for all analytes of interest. MDLs shall be determined as follows:

- The laboratory shall prepare a standard matrix sample at 1 to 5 times the estimated MDL (based on the RDL and the instrumental detection limit);

- 7 aliquots of the sample shall be processed through the entire method;

- The standard deviation shall be calculated from the results of the seven aliquots;

- The MDL is equal to the standard deviation times the Student's t value (3.143) for that number of measurements.

The MDL shall be equal to or less than the Required Detection Level (RDL). If the calculated MDL is lower than what the laboratory considers a practical detection level then the MDL may be raised to the higher level. In no instance shall the MDL be lowered below the calculated level. The method documentation (section 5.2.3) shall include both the calculated MDL and the request for an increased MDL. MDLs for inorganics shall be verified quarterly. MDLs for organics shall be verified annually.

If the laboratory has verified an MDL within the timeframes specified above, it shall not be necessary for the laboratory to repeat the verification process.

This procedure is based upon 40 CFR Chapter 1, and upon the CLP inorganic Statement of Work.

All data related to determination and verification of MDLs shall be maintained at the laboratory.

5.2.3 ANALYTICAL METHODS DOCUMENTATION

An analytical method shall be described by a set of written instructions (Method Documentation Package) citing the basic method (ie. CLP or SW-846), any changes to the basic method, descriptions of analytes, sample type (matrix), MDLs and Upper Reporting Levels (URLs), and calibration standard levels, and a copy of the calibration curve used for the MDL determination. The package shall also include the laboratory SOP for the basic method and details of the preparation of **all** calibration and spiking solutions, from stocks to working standards. MDLs shall be determined as specified in Section 5.2. The URL shall be the highest value which the laboratory can report and to which the method is calibrated. The laboratory shall specify how the URL was selected. All values above the URL shall be diluted to within the reporting range. When the basic method offers a choice of options the method shall specify which option(s) was selected. The analytical method shall be followed throughout the entire project. The Method Documentation Package shall be submitted to the USAEC Chemistry Branch for approval through the COR/project officer. After approval of a method, additional deviations shall not be acceptable, unless written approval, in advance, is provided by the USAEC Chemistry Branch through the COR/project officer. In urgent cases verbal approval may be granted, however, this must be immediately followed by a written approval. Any change in the documented procedure

shall constitute a modification. The significance of the modification will be determined by the USAEC Chemistry Branch. Changes made after approval may require generation of new MDLs. Any method that offers the capability for analyte confirmation (e.g., second column confirmation for a GC method) shall have the confirmation procedure included as part of the method writeup. Determination of the MDL shall also be required for the confirmation procedure. If the Confirmation MDL is greater than the Method MDL the USAEC Chemistry Branch will decide if the results are acceptable on a case by case basis. If a method has the capability to use both columns for quantitation, then the same column shall always be used for a given compound. The column to be used for quantitation shall be specified in the method documentation package.

Methods specifically designated as Field Detection Methods should also follow the requirements of validation as described in these Guidelines and contain the necessary statements/procedures for the associated QA/QC.

5.3 METHODS NOT REQUIRING VALIDATION

Some methods, including calibration of test and measurement equipment, do not require validation, due to either the nature of the measurement or the intended use of the data. When such methods are part of a project, the USAEC will not provide a standardized method. However, laboratories must submit sufficient information in test plans, work plans, project QC plans, etc., to describe exactly the procedures to be used. A copy of the methods must be submitted to the USAEC before it is used on any project.

The following methods do not require validation:

- Temperature;
- Conductivity;
- pH;
- Oil and Grease;
- Hardness;

- Asbestos;
- Alkalinity, Carbonate/Bicarbonate/Hydroxide;
- Total Organic Carbon (TOC);
- Biochemical Oxygen Demand (BOD);
- Chemical Oxygen Demand (COD);
- Total Dissolved Solids (TDS);
- Total Suspended Solids (TSS);
- Salinity;
- Total Solids;
- Acidity;
- Total organic Halogen (TOX); and
- Dissolved organic carbon (DOC).

Other methods that may be included in this category should be brought to the attention of the Chemistry Branch for consideration.

5.4 METHOD DEVELOPMENT

In the event that analyses must be conducted for compounds for which no reliable methods exist, development of a method will be conducted by a Development Laboratory (laboratory designated to develop an analytical method). The Development Laboratory may be a contractor laboratory tasked to perform the development, or it may be a government laboratory. Documentation for Proposed Methods Development (Appendix A) shall be submitted to the USAEC Chemistry Branch for approval prior to initiation of method development.

The Chemistry Branch will evaluate the proposed approach for technical soundness and economy of effort. The Chemistry Branch will then request the Development Laboratory to proceed with the method development, either as proposed or with USAEC recommended modifications.

The Development Laboratory shall investigate the proposed procedures to be included in the method. Should any of the proposed procedures approved by the Chemistry Branch be found to be inadequate for the method, alternative procedures will be investigated after approval by the Chemistry Branch.

When testing of the analytical procedures has been successfully completed by the Development Laboratory, the method shall be fully documented.

Full documentation of the method shall be submitted to the USAEC Chemistry Branch. The Chemistry Branch will review the documentation for completeness and comprehension. Based on this review, the Development Laboratory will make any necessary modifications. After final approval by the Chemistry Branch, the method will be issued as a final method. Chemistry Branch shall inform MRD of method development initiatives.

6.0 GENERAL LABORATORY PROCEDURES

6.1 STANDING OPERATING PROCEDURES - LABORATORY

The laboratory shall have written SOPs for all procedures and methods, including sample analysis, laboratory functions, and auxiliary functions, prior to the analysis of field samples. Procedures and methods shall be performed in the laboratory as described in the SOP. Any modification of an SOP made during a data collection activity must be documented and approved in writing by the USAEC Chemistry Branch through the COR/project officer. SOPs shall be prepared for, but not limited to, those listed in Appendix G.

A description of the basic information required in each of the above SOPs is included in Appendix G. The laboratory SOP is not required to conform to a specific format but shall be representative of standard laboratory operations, and shall give clear evidence of the laboratory's ability to successfully fulfill all contract requirements.

6.2 LABORATORY PERSONNEL GUIDELINES

Guidelines to be used in the determination of personnel qualifications are as follows:

- Laboratory Director - should have earned a Baccalaureate Degree in Science or Engineering from an accredited college or university or the equivalent and have at least 5 years experience in laboratory work.

- Senior Staff - should have earned a Baccalaureate Degree in Science or Engineering from an accredited college or university or the equivalent and have at least 2 years experience at the bench level.

- Technical Staff - should have formal training in the sampling and analytical methodology and quality control as applied to the specific sample types and concentration levels of analytes which are of interest to the project.

These requirements are based upon those contained in the CLP Statements of Work.

6.3 USAEC METHOD CLASSES

USAEC divides analytical methods into 4 classes for determining the number and types of QC samples per lot, and for use in automated data validation routines. The USAEC method classes are as follows:

- CLASS 1 Methods - These are methods for the analysis of organic parameters, with the exception of GC/MS methods and pesticides/PCBs by GC, and for the analysis of inorganic parameters.

- CLASS 1M Methods - These are GC/MS methods, both for the analysis of volatiles and semivolatiles.

- CLASS 1P Methods - This class is restricted to methods for the analysis of pesticides and PCBs by GC.

- CLASS 2 Methods - This class is reserved for screening type methods, which give only a qualitative (i.e. yes/no) result.

6.4 USAEC SAMPLE IDENTIFICATION NUMBERS

The reporting of analytical results to the USAEC Installation Restoration Data Management Information System (IRDMIS) requires that each sample aliquot be assigned a unique seven character identification number. The first four characters of this number are alpha characters that represent the analytical lot. Each analytical lot is given a different series of alpha characters. For instance a group of water samples for Metals analyses by ICP could be assigned the alpha designation of AAAA. Another group of samples that contain samples for Anion analyses, some to be done by Technicon and others to be done by IC, would be given two different alpha designations. The Technicon analyses could be given a designation such as AAAB and the IC analyses could be given a designation such as AAAC. In the case of a multi-analyte method, the alpha designator assigned will be the same for each analyte

in a single sample aliquot.

The last three characters are numeric characters that represent the individual samples within the lot. The lot size must be determined before these numbers can be assigned. The lot size is defined as the number of samples that can be extracted, analyzed, or digested in a single day as controlled by the rate limiting step in the particular method (see Section 6.9). When USAEC approves a particular method write-up during the validation process, it also approves a lot size.

If the contractor laboratory uses an internal numbering system a correlation of the internal lab sample number to the USAEC lot number shall be recorded in a bound logbook.

6.5 SAMPLE HOLDING TIMES

The time that a preserved sample may be held between sampling and analysis is based on the analyte(s) of interest. Holding time limitations are intended to minimize chemical change in a sample before it is analyzed. The holding time is the maximum time allowable between sample collection and the completion of analysis, based on stability factors. The holding times specified in this document do not preclude shorter analysis and reporting requirements which may be specified in the contract. Allowable holding times (Table 6-1) apply to both solid and aqueous samples. Results reported for samples analyzed after holding times have been exceeded shall normally be considered out-of-control and unacceptable. To expedite analysis and to minimize the possibility of exceeding holding times, samples should be sent to the laboratory by an overnight courier service, as soon as possible after collection. The holding times specified in Table 6-1 are based on the most restrictive holding times required by the EPA and do not necessarily match the holding times in ER 1110-1-263.

TABLE 6-1 REQUIRED HOLDING TIMES FOR USAEC SAMPLES

Analysis	Holding Time
Volatiles - Aqueous	14 Days Preserved 7 Days Unpreserved
Volatiles - Solid	14 Days (Some EPA Regions may only allow 10 days)
Semivolatiles - Aqueous/ Solid	7 Days for Extraction 40 Days for Analysis
Pesticides/PCBs - Aqueous/ Solid	7 Days for Extraction 40 Days for Analysis
Explosives - Aqueous/ Solid	7 Days for Extraction 40 Days for Analysis
Cyanide - Aqueous/Solid	14 Days
Mercury - Aqueous/Solid	28 Days
Metals (except Mercury) Aqueous/Solid	180 Days
Anions - Aqueous/Solid	28 Days (48 Hrs for NO2/NO3 speciation)
TPHC - Aqueous/Solid	28 Days
CR(VI) - Aqueous/Solid	24 Hrs

6.6 STANDARD WATER SAMPLES

Standard water samples shall be used for standard matrix quality control spikes. Standard water samples will be prepared by adding a known quantity of target analyte to a known volume of water. The volume of water will be specified in the method being performed. All control analytes for the method will be added. ASTM Type I grade water will be used for inorganic methods (Table 6-2). ASTM Type II grade water containing 100 mg/L each of added sulfate and chloride will be used for organic methods (Table 6-2). The method and reagents used to prepare spiking solutions are specified in the standardized methods.

6.7 STANDARD SOIL SAMPLES

USAEC supplied standard soil shall be used for standard matrix quality control spikes. Standard soil samples will be prepared by adding a known quantity of the control analyte to a known weight of selectively blended standard soil as provided by the Chemistry Branch. This standard soil is provided to the Contractor Laboratory after contract award. The required amount of soil (sample weight) to be spiked will be specified in the method being tested. A minimum quantity of solvent shall be used so that the character of the sample is not changed. The normal solvent to soil ratio is 1:2 (ie. 5 ml for a 10 g sample). All control analytes for the method will be added. With the exception of volatiles, spikes must sit in contact with the soil for a minimum of 1 hour before processing of the sample continues. Spikes for volatiles shall be analyzed immediately following the spiking procedure. The method and reagents used to prepare spiking solutions are specified in the standardized methods. The Contractor Laboratory will be provided a sufficient quantity of this standard soil to last for the duration of the project or series of projects.

Table 6-2. CRITERIA FOR ASTM WATER TYPES

Grade of Water	Maximum Total Matter (mg/L)	Maximum Electrical Conductivity at 25C (umho/cm)	Minimum Electrical Resistivity at 25C (M cm)	Minimum Color Retention Time of $KMnO_4$ (min)
Type I	0.1	0.06	16.67	60
*Type II	0.1	1.0	1.0	60

* 100 mg/L Sulfate and Chloride Added. The following preparation is provided:

(1) Weigh 1.48 g of reagent grade anhydrous sodium sulfate into a 1-liter volumetric flask and dilute to mark with ASTM Type II water.

(2) Weigh 1.65 g of reagent grade anhydrous sodium chloride into a 1-liter volumetric flask and dilute to mark with ASTM Type II water.

(3) Transfer 100 ml of each solution prepared in (1) and (2) into a 1-liter flask and dilute to volume with ASTM Type II water.

6.8 SAMPLE PREPARATION/FILTRATION

Water used in the course of organic analyses shall conform to ASTM Type II grade, as defined in Table 6-2. Water used in the course of inorganic analyses shall conform to ASTM Type I grade, as defined in Table 6-2. Standard and QC samples for organic analyses shall be prepared with water which conforms to ASTM Type II grade with 100 mg/L sulfate and chloride added.

6.8.1 WATER SAMPLES

The need to filter water samples depends on whether total or dissolved contaminants are of interest. The project-specific decision must be explicitly stated in the Quality Assurance Project Plan. Assessment objectives must be considered when specifying filtration requirements, procedures, and materials in the Project Workplan.

Samples for any dissolved constituents (organic or inorganic) must be filtered in the field if a chemical additive is used for preservation. Volatile organic compounds and oil/grease are the only universal exemptions to this guideline; samples for these two analyte classes are never filtered. Samples for dissolved metals analyses must be filtered in the field, before adding chemical preservatives, to preclude extraction of contaminants from the particulate matter by the preservatives. Samples for organic analyses generally should be filtered in the laboratory. The filter material used in the field or the laboratory must be compatible with the constituents of interest. Compatibility is defined in the following way:

- The filter material is not changed by the material being filtered (and vice versa); and

- The filter material does not absorb or leach the chemical species for which the sample will be analyzed.

The compatibility requirement may necessitate filtering individual subsamples for specific analytes if a universally compatible filter material cannot be identified. Exceptions to these guidelines must be obtained in writing from the USAEC Chemistry Branch. However, if the proposed filter material(s) meet the above requirements, no additional approval is required.

6.8.2 SOIL/SEDIMENT SAMPLES

Soils and sediments are very complex mixtures with widely varying compositions, even within a single site. Recovery of analytes depends on many factors, including organic content, mineral content, particle size, and moisture content of the soil. Soil and sediment samples shall be analyzed in the as-received condition and prepared as follows:

- The sample shall be mixed as thoroughly as possible in the wide-mouth, amber-glass bottle by shaking and/or stirring. Glass or Teflon rods may be used for stirring (does not apply to samples for volatiles analysis, as no mixing may be performed on these samples).

- For each sample, an aliquot of the as-received sample shall be dried according to the procedure in ASTM D2216-71, "Laboratory Determination of Moisture Content of Soil" (Note that the calculations specified in the method do not apply; only the drying procedure itself is of interest). The calculated percent moisture for each sample shall be entered into the USAEC IRDMIS as described in Section 9.4. The determination of percent moisture is calculated as follows:

$$\frac{\text{Sample Weight (wet)} - \text{Sample Weight (dry)}}{\text{Sample Weight (wet)}} \times 100$$

- The moisture determination on a sample designated for volatiles analysis shall be performed on a duplicate of the sample and not the sample itself.

- Weighed aliquots of the mixed sample shall be obtained for each analysis. All samples will be analyzed and reported in the as-received condition.

6.9 SAMPLE ANALYSIS/LOTS

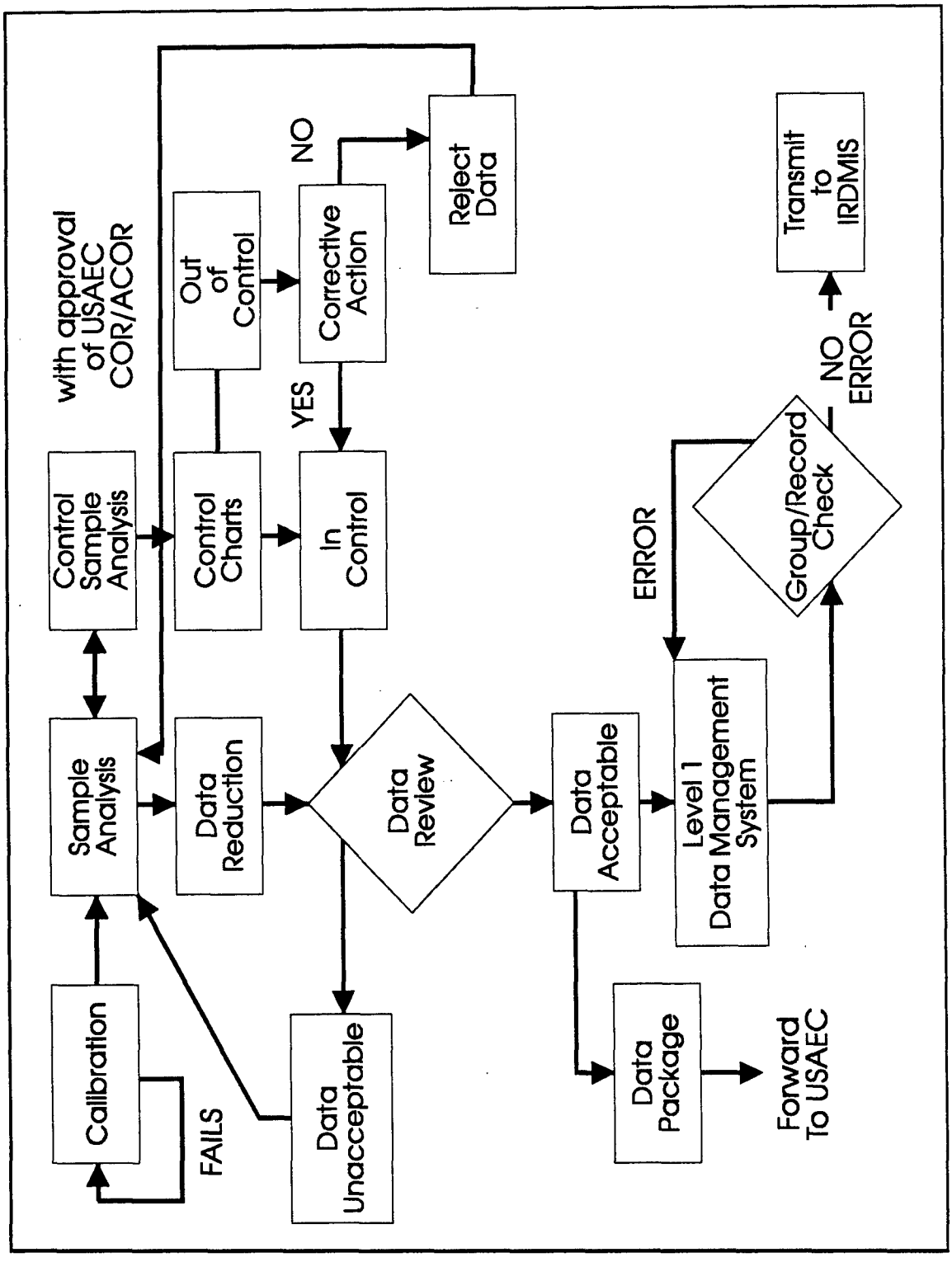
All samples shall be analyzed by lot. A lot is the maximum number of samples, including QC samples, that can be processed through the rate limiting step of the method during a single time period, not to exceed one 24 hour day, except for Class 1P methods, where the instrumental analysis may continue for 48 hours. The time period for a lot does not include the initial or daily lot calibrations, provided that sample analysis begins immediately following completion of the calibration. Analysis of samples within a lot must be as nearly continuous as possible. Lots shall not be mixed; that is, all samples for one lot, including QC samples, must be completed prior to the beginning of a new lot. Any break in the analytical sequence shall not exceed 2 times the analytical run time and must be fully documented. For methods with multi-step extractions, each subsequent step must begin immediately, or on the next normal business day. The rate of sample collection or shipment does not determine maximum lot size, although it may limit the number of samples available for analysis at a given time. A lot may consist of samples from more than one installation as long as the data quality objectives for each of the installations are the same. All samples in one lot must be completely processed through any given step in the same time period. For example, suppose a laboratory can extract 10 samples at one time, can concentrate 20 sample extracts at a time, and can instrumentally analyze 50 sample extracts at a time. The lot may only contain 10 samples because no more than 10 samples can be processed at one time during the rate limiting step, in this case the extraction step.

All samples must be processed through the entire analytical method, exactly as validated. Any proposed modifications to the validated method must be evaluated and approved by the USAEC Chemistry Branch through the COR/project officer before use. Any field samples with concentrations of any analyte above the URL shall be diluted within range for concentration measurement (QC samples are never to be diluted). Records of all dilutions must be maintained and the dilution factors shall be entered into the USAEC IRDMIS (Section 9.4). The method of analyte identification and quantification will be specified in the analytical methods. A typical sequence of sample analysis through data transmission is shown in Figure 6-1.

In any chromatographic method, excluding GC/MS and ion chromatography, the presence of a compound shall be confirmed (as long as confirmatory method is available) on a second column. Confirmation does not necessarily have to be

performed within holding times but must be accomplished within 10 days of sample analysis. Results of confirmatory analyses must be reported with the original data within the time specified by the contract or task order.

Figure 6-1.


Calibration
FAILS
Sample Analysis
Control Sample Analysis
with approval of USAEC COR/ACOR
Data Reduction
Control Charts
Out of Control
NO
Corrective Action
YES
In Control
Reject Data
Data Review
Data Unacceptable
Data Acceptable
Data Package
Forward To USAEC
Level 1 Data Management System
ERROR
Group/Record Check
NO ERROR
Transmit to IRDMIS

6.10 INSTRUMENT MAINTENANCE

This section establishes procedures for maintaining test and measurement equipment used to conduct analyses, in such areas as instrument maintenance, service contracts, and absolute physical or electronic calibration. Chemical calibration is discussed in Section 7.0.

The calibration policies and procedures set forth will apply to all test and measuring equipment. All test and measuring instruments fall into two general categories: those which are calibrated prior to each use and those which are calibrated on a scheduled, periodic basis.

All equipment to be calibrated will have an assigned record number permanently affixed to the instrument. A label will be affixed to each instrument showing: description, manufacturer, model number, serial number, date of last calibration or maintenance, by whom calibrated/maintained, and due date of next servicing. Calibration reports and compensation or correction figures will be maintained with the instrument. Thermometers are exempt from the labeling requirement, but not from the calibration requirement.

A written stepwise calibration procedure must be available for each piece of test and measurement equipment. Any instrument which is not calibrated to within the manufacturer's original specifications must display a red warning tag to alert the analyst that the device carries only a "limited calibration." Equipment unable to meet approved calibration specifications shall not be used for sample analysis.

It is the contractor's responsibility to maintain an adequate supply of critical spare parts to minimize instrument down-times.

6.11 CALIBRATION IDENTIFICATION

Instruments past due for calibration or maintenance must be immediately removed from service, either physically or, if this is impractical, by tagging, sealing, labeling, or other means.

The labeling and recording system extends to calibration or maintenance services provided to the Contractor Laboratory by other organizations. Certifications and reports furnished by these organizations should be filed and made a part of the required record keeping system.

Equipment in "Calibrate Before Use" (CBU) status must be administratively sequestered to avoid accidental use without calibration.

6.11.1 CALIBRATION STANDARDS

All physical or electronic measurements or calibrations (excluding chemical calibration curves) performed by or for the Contractor Laboratory must be traceable, directly or indirectly, through an unbroken chain of properly conducted calibrations (supported by reports or data sheets) to the NIST. Reports must be up-to-date for each reference standard and each subordinate standard used for calibration of test and measurement equipment. When calibration services are performed by a non-contractor laboratory organization, copies of reports, and records showing traceability to the NIST should be immediately available. These records may be inspected during laboratory audits.

6.11.2 CALIBRATION FREQUENCY SCHEDULE

At a minimum, calibration and maintenance intervals for complex or sensitive laboratory instruments must be those recommended by the respective manufacturers, unless experience dictates a shorter interval. When the manufacturer has not specified a calibration interval for its equipment, the interval will be established in writing by the calibration group servicing the laboratory. Adherence to the schedule is

mandatory. The fact that these checks may be scheduled and performed by an outside source does not exempt the laboratory from its responsibility for identifying, monitoring and controlling calibration intervals, and ensuring that checks are made on time.

6.11.3 EXAMPLES

Routine, "absolute" calibration is not the same as chemical calibration, where the relationship between instrument response and concentration is established. "Absolute" calibration ensures that the perceived instrument response corresponds to the correct physical signal that should produce that response. Examples of equipment that must be "absolutely" calibrated include, but may not be limited to, the following:

- Balances -- These are the clearest examples of equipment requiring calibration. NIST-certified weights are used to ensure the accuracy of measurements.

- Thermometers -- NIST-certified thermometers are used to verify the accuracy of measurements.

- Other Temperature Sensors and Controllers -- For analytical equipment that incorporates temperature sensing or control, the accuracy of the sensors and controllers will affect method performance. When a method specifies an injector temperature of 100°C, the analyst must be sure that the instrument settings for 100°C actually corresponds to that temperature. Oven temperatures (e.g., drying ovens, GC ovens) must be accurately known. Equipment manufacturers describe procedures for temperature calibration, using either NIST-calibrated thermometers or measured electrical signals.

- Flow Controllers -- Measuring and controlling gas and liquid flow are integral parts of many instrumental analysis systems. The devices used to measure/control must be calibrated to ensure that actual flow corresponds to instrument readings or settings. ICP, IC, GC, GC/MS, and HPLC are examples of systems that must be calibrated for flow.

- Autoinjectors -- The actual volume injected into the analytical system must correspond to the instrumental settings for the intended volume. This calibration is particularly critical when absolute analyte response (e.g., peak height) is used for

quantification (as opposed to the ratio of analyte peak height to internal standard peak height).

- Recorders -- When physical records (e.g., strip charts) are used for quantification, the recorder response must correspond to the electronic signal received. If the basis of quantification is a linear relationship between response and concentration, the recorder must exhibit linear response to linear changes in electric signals.

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7.0 CALIBRATION REQUIREMENTS

7.1 CHEMICAL CALIBRATION CURVES

Before samples are analyzed on an instrument, chemical calibration standards of each target analyte must be analyzed to establish that the instrument is functioning properly with the desired sensitivity. Economy of effort dictates that as many analytes as possible be combined in the chemical calibration standards.

Chemical instrument calibration shall be accomplished using calibration standards prepared by mixing the species to be analyzed in the solvent that is introduced into the instrument, as dictated by the analytical method. The concentrations of the chemical calibration standards will be chosen to cover the allowable reporting range of the method. That is, at least one calibration standard will have a concentration equal to the MDL and at least one calibration standard will have a concentration equal to the upper reporting limit.

Data from the chemical calibration standards shall be plotted with the instrument response indicated on the ordinate and the concentration indicated on the abscissa. When microprocessors are used to establish calibration curves, the data must nevertheless be plotted. If, after plotting, the curve is shown to be linear with acceptable variance, the microprocessor may be used to determine analyte concentrations in samples. Methods and formulae for quantification shall be as specified in the standardized methods.

Chemical instrument calibration curves shall not be used to determine the MDL. Rather, analysis of chemical calibration standards are to be used by instrument operators to establish response versus concentration relationships and to provide early warning of instrument variances.

Data from the calibration checks are to be recorded on forms (Appendix R) and maintained with the lot data package. Alternatively, if a laboratory-wide computerized data management system is available, data calibration may be generated electronically and output on forms or charts. In either case, documentation must be available to demonstrate the validity of the calibration checks.

7.1.1 INITIAL CALIBRATION, CLASS 1, CLASS 1P, AND CLASS 1M METHODS

Initial Calibration procedures shall be used whenever:

- The method detection level (MDL) is determined;
- The instrument is started up (other than daily start up and shut down);
- The instrument is used to analyze analytes different from those for which the instrument was previously calibrated; and
- The instrument fails daily or continuing calibration.

Initial calibration shall be as specified in the analytical method, however, in addition, one standard at the MDL and one standard at the URL shall be analyzed. If no calibration requirements are specified in the method, then refer to the USAEC Chemistry Branch for guidance. The concentrations of the calibration standards, in the solvent that results from all the preparation steps of the method, shall take into account any concentration steps that are part of the method. Concentrations in the solvent shall correspond to those in an environmental matrix as if the method preparation steps had been performed.

In addition to the initial calibration standards, Class 1 and 1P methods require the analysis of calibration check standards (Section 7.4). During a Class 1 or Class 1P initial calibration, a calibration check standard shall be analyzed at the completion of calibration. If the method requires what could be an initial calibration each day analysis is performed, then the calibration check standards are to be analyzed once a week rather than each day. The concentration of the calibration check standard shall be near the upper end of the Method Reporting Range (MRR) and shall contain all the analytes of interest. Calibration check standard results shall be within the limits of acceptability defined in Sections 7.4 and 7.5.

If the results of the calibration check standard are not acceptable, immediate reanalysis of the calibration check standard is required. If the results of the reanalysis still exceed the limits of acceptability the system is considered to have failed calibration. Sample analysis shall be halted and shall not resume until successful completion of initial calibration. Corrective action(s) taken to restore initial calibration shall be documented by the contractor laboratory.

7.1.2 DAILY LOT CALIBRATION, CLASS 1, CLASS 1P, AND CLASS 1M METHODS

Calibration standards shall be analyzed at the start of each lot, prior to sample analysis, to verify that instrument response has not changed from previous calibration. Daily lot calibration shall be performed in accordance with the requirements of the analytical method. If no calibration requirements are specified in the method, then contact the USAEC Chemistry Branch for guidance. **NOTE: For pesticides/PCBs it is suggested that the daily lot calibration consist of CLP Mix A, CLP Mix B, Toxaphene, and PCBs 1016 and 1260.** The response of the daily lot calibration must fall within the limits of acceptability as defined in section 7.5. If the response fails this test, the daily lot calibration shall be reanalyzed. If the response from the second analysis is not within the limits of acceptability, Initial Calibration must be performed before analyzing samples.

After sample analyses are completed for the lot an ending daily lot calibration standard, as specified in the method, shall be analyzed. If the response is not within the limits of acceptability, the daily lot calibration standard shall be reanalyzed. If the response from the second analysis is not within the limits of acceptability, the system is considered to have failed calibration. Initial Calibration must be performed and all samples analyzed since the last acceptable calibration must be reanalyzed. Note that the ending daily lot calibration may also serve as the beginning daily lot calibration for a subsequent lot, provided that there is no break in the analytical sequence.

For both the beginning and ending calibrations, if the first attempt fails to meet criteria, minor maintenance (ie. snipping the end of the GC column, cleaning the injection port, etc.) may be performed. All such activities shall be documented.

In addition, a special case exists for beginning calibrations with no preceding

analyses (ie. there has been a break since the last analysis by this method). If the second attempt to calibrate fails, then the laboratory may prepare a new daily calibration standard and reanalyze. If this third attempt fails then initial calibration shall be performed.

7.1.3 CONTINUING CALIBRATIONS

Continuing calibration, in accordance with the EPA CLP Statement of Work, shall be performed as follows:

- For inorganics, a blank and a continuing calibration standard shall be analyzed after every 10th sample, or every 2 hours, whichever is more frequent. The standard shall be near the mid-point of the method reporting range and shall meet the limits of acceptability as specified in Section 7.5.

- For GC/MS volatiles, a blank and a continuing calibration standard shall be analyzed every 12 hours. The standard shall meet the limits of acceptability as defined in Section 7.5.

- For GC/MS semivolatiles, a continuing calibration standard shall be analyzed every 12 hours. The standard shall meet the limits of acceptability as defined in Section 7.5.

- For pesticides and PCBs, the laboratory shall analyze a blank every 12 hours. In addition, every 12 hours the laboratory shall alternately analyze a Performance Evaluation Mixture (PEM) or Standard Mixtures A and B as defined in the EPA CLP requirements. All results for the PEM and Standards shall meet the limits of acceptability as defined in Section 7.5.

- For all other organic methods, the laboratory shall analyze a blank and a continuing calibration standard every 12 hours. The standard shall meet the limits of acceptability as defined in Section 7.5.

- If a continuing calibration fails to meet the limits of acceptability the laboratory shall immediately reanalyze the standard. If the first analysis fails to meet criteria, minor maintenance (ie. snipping the end of the GC column, cleaning the injection port, etc.) may be performed. All such activities shall be documented. If this reanalysis is

not acceptable then all analyses shall cease until the cause of the problem is determined and corrected. All samples analyzed since the last acceptable calibration shall be reanalyzed.

7.1.4 INITIAL CALIBRATION, CLASS 2 METHODS

The instances when Initial Calibration must be performed are the same as described in Section 7.1.1. Calibration standards shall be prepared and analyzed in triplicate at concentrations of 0 (blank) and the MDL. The spiked concentration shall correspond to the MDL in the environmental matrix. All blanks must yield negative results and all spiked samples must yield positive results for acceptable calibration.

7.1.5 DAILY LOT CALIBRATION, CLASS 2 METHODS

Before and after sample analysis of each lot, one blank and one calibration standard at the MDL shall be analyzed. If any calibration standard yields an inappropriate response (positive for a blank, or negative for the spiked standard), a second calibration standard shall be analyzed. If the second standard yields an inappropriate response, the system is considered to have failed calibration. The cause of the failure must be determined and corrected before analyses may continue.

If calibration failure occurs at the end of sample analyses, the analytical results obtained since the last satisfactory calibration are considered invalid and must be repeated. After calibration failure, the procedure for the Initial Calibration must be followed to demonstrate satisfactory performance.

SECTION 7.2 GC/MS TUNING

All GC/MS methods shall require the instrument to be tuned every 12 hours while in operation. When analyzing volatiles, bromofluorobenzene shall be used to tune the instrument, while decafluorotriphenyl phosphine shall be used for semivolatile analyses. These requirements, and the criteria for acceptability shall be as specified in the latest EPA CLP requirements.

SECTION 7.3 ICP METHOD SPECIFIC REQUIREMENTS

For all analyses conducted by ICP the following requirements from the CLP Statement of Work shall be met:

- Interelement correction factors shall be calculated annually, or whenever major maintenance is performed on the instrument.

- Interference check samples shall be run twice per lot or twice per 8 hours, whichever is more frequent.

SECTION 7.4 CALIBRATION CHECK STANDARDS

SECTION 7.4.1 REQUIREMENTS FOR USE

Calibration check standards are required for all Class 1 and 1P methods and shall be analyzed with each initial calibration. If an initial calibration is performed each day then the calibration check standard shall be analyzed once per week (once per five lots if analyses are not performed daily). The calibration check standard shall contain all analytes of interest for the method in question at a concentration near the upper end of the calibration range.

SECTION 7.4.2 SOURCES OF CHECK STANDARDS

CASE 1. A certified check standard is available from the EPA or some other source with both the true value and limits of acceptability specified by the supplier. The results must fall within the limits specified in Section 7.5 or by the supplier, whichever is less.

CASE 2. A certified check standard is available from the EPA or some other source with a true value specified but without limits of acceptability. The results must fall within the limits specified in Section 7.5.

CASE 3. If no certified check standard is available, the contractor laboratory shall prepare a check standard using a second source of reference material. This standard shall be prepared by a different analyst than the one who prepared the calibration standard. If weighing of the material is required, a different balance should be used, if possible. The results must fall within the limits specified in Section 7.5.

CASE 4. If there is only one source of reference material available, then the calibration and calibration check standards must be prepared from the same material. The standards shall be prepared by different analysts. If weighing is required, different balances should be used, if possible. The results must fall within the limits specified in Section 7.5.

7.5 LIMITS OF ACCEPTABILITY

Limits of acceptability are based upon those contained in the EPA CLP Statements of Work.

7.5.1 INORGANICS

- All metals except mercury shall be within +/- 10%.
- Mercury shall be within +/- 20%.
- Anions shall be within +/- 15%.
- All other inorganics shall be within +/- 15%

7.5.2 ORGANICS

- For GC/MS methods, 2/3 of the analytes shall be within +/- 25%, and all analytes shall be within +/- 40%.
- For all non-GC/MS organic methods all analytes shall be within +/- 25%.

• When analyzing the PEM for pesticides/PCBs the breakdown of DDT and Endrin shall be less than 20%, and the combined breakdown of DDT and Endrin shall be less than 30%.

• When response factors are used the daily and continuing standards shall be compared to the average response factor of the initial calibration.

7.6 REFERENCE MATERIAL

During chemical calibration and sample analyses, solutions containing known analytes at known concentrations must be prepared. These solutions are needed to generate method performance data, calibrate instruments, spike analytical surrogates or internal standards, prepare QC samples, and prepare performance samples, when specified. Three types of reference materials may be used to prepare standard solutions, as described in Sections 7.6.1 through 7.6.3.

Before initiating any laboratory studies, the Contractor Laboratory must submit a request to the USAEC Project Officer or Contracting Officer's Representative for reference materials. The list should include all target analytes of interest on a specific project, surrogate compounds, and internal standards. The USAEC Project Officer or Contracting Officer's Representative will forward the request to the USAEC Chemistry Branch. Samples of reference materials will be shipped to the Contractor Laboratory from the repository. Only if reference materials are not available through USAEC should the Contractor Laboratory obtain the materials from an outside source.

Reference materials for metals and non-metallic inorganics may be maintained at room temperature in a locked storage area. All other reference materials must be stored in a locked refrigerator at or below 4°C. All reference materials shall be maintained under chain-of-custody. An SOP for the use, control, and inventory of reference materials will be prepared.

7.6.1 STANDARD ANALYTICAL REFERENCE MATERIALS (SARMs)

Whenever possible, chemical analyses conducted in support of USAEC projects should be based on SARMs. These materials are labeled as SARMs and carry a SARM identification number. These materials will either be National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs) or will be traceable to NIST SRMs. The SARM Repository Program is described in Appendix F. Contractors are encouraged to use secondary standards that are referenced to SARMs and are periodically checked against SARMs. This check will be performed the first time the standard is used and at six month intervals or when the standard is replaced, whichever comes first. The use of secondary standards are encouraged as a conservation method for the more costly SARMs.

7.6.2 INTERIM REFERENCE MATERIALS (ITRMs)

ITRMs are available from two sources. Some of these materials are maintained and distributed by the USAEC and should be used if SARMs are not available. Although ITRMs are supplied through the USAEC, they are not as rigorously characterized, as are SARMs. ITRM characterization includes positive identification of the material and an estimate of purity. The SARM label on each bottle is modified by adding the word "Interim" and includes an identification number. These materials may be used as received from the USAEC. Reference materials obtained from the U.S. Environmental Protection Agency, or NIST do not require characterization by the Contractor Laboratory.

7.6.3 OFF-THE-SHELF MATERIALS

SARMs or ITRMs may not be available for some target analytes. If materials are unavailable through USAEC, Contractor Laboratories will be instructed to purchase materials from an outside supplier. These materials shall be considered as "off-the-shelf." Before using any material, regardless of source, classified as "off-the-shelf," the Contractor Laboratory must analyze the material to obtain a positive identification and estimate of purity. Where possible, characterization analyses for purity shall be conducted using at least two different methods. Off-the-shelf materials should be

compared to NIST or EPA standard material whenever possible. The characterization analyses must be performed before method validation is initiated and the results must be provided to USAEC with the Method Documentation Package. Documentation for purity and identity characterization analyses shall be kept on file at the contractor laboratory. Possible techniques for characterizing the off-the-shelf materials include, as applicable:

- Infrared spectroscopy;
- Melting point, decomposition point, or boiling point determinations;
- Mass spectrometry;
- NMR spectrometry;
- Elemental analysis;
- Gas chromatography (for purity); or
- Liquid chromatography (for purity).

This list is not exhaustive and all of the listed techniques need not be used. The Contractor Laboratory is responsible for providing positive identification and a purity estimate for each off-the-shelf material (including internal standards) to USAEC.

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8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 INTRODUCTION

In addition to the requirements discussed thus far, QC samples must be analyzed to provide quantitative evidence that the entire method is performing acceptably. It is essential that controls are initiated during and maintained throughout the analysis of samples. Data generated from the control samples are plotted on control charts, which are used to monitor day-to-day variations in routine analyses.

For multi-analyte methods, the selection of control analytes will be specified at the time the method is approved. As a rule, no less than 50 percent of the target analytes will be selected as control analytes, with the minimum number selected being 4. That is, for any method having four or fewer target analytes, all analytes will be selected as control analytes; for a method having 5 to 8 target analytes, 4 will be selected as control analytes; for each additional 1 or 2 target analytes, 1 additional control analyte will be selected. Exceptions will be specified in the appropriate standard method. Note that this rule does not apply to GC/MS or ICP methods.

For GC/MS methods, only the surrogates will be used as control analytes. The surrogates used shall be as specified in the method. For ICP water methods, due to interelement factors, all analytes must be spiked into the control samples. However, only those analytes which are to be reported need be control charted. For ICP soil methods the same rule applies, however, Al, Ba, Ca, Fe, Mg, Mn, K, and Na shall not be used as spike elements.

Note that USAEC does not normally require the matrix spikes and matrix spike duplicates required by the EPA. The EPA uses these samples to determine matrix effects and within day variability of the laboratory. In lieu of these, USAEC will perform the following:

- Matrix effects shall be determined using surrogates in each field sample, if appropriate surrogates are available. If surrogates are not available, then matrix spikes will be performed at a rate of 1 per 20 samples.

- The within day variability of the method will be determined using the USAEC

required duplicate, standard matrix, QC spikes. This will replace the matrix spike duplicates.

8.2 CONTROL SAMPLES

Control samples are those samples that are introduced into the train of environmental samples to function as monitors on the performance of the analytical method. All required QC samples shall be prepared from standard matrices (Sections 6.6 and 6.7) or actual field samples, as required, and processed through the complete analytical method. Stock solutions used to spike QC samples shall be prepared independently of stocks used for calibration standards.

Numbers and concentrations of QC samples required for different method classes, per lot of field samples, are summarized in Table 8-1. The analysis sequence for Class 1P control samples shall be as specified in Table 8-2. For Class 1M method, if the lot requires more than 12 hours for analysis then one QC sample shall be analyzed in each 12 hour period.

Routine reanalysis of QC samples is not permitted. Justification for reanalysis of QC samples must be fully documented.

8.2.1 TYPES OF CONTROL SAMPLES

The following types of QC samples shall be included in each analytical lot:

Class 1 Methods:

- Method Blank, to verify that the laboratory is not a source of sample contamination; and

- Spikes of all control analytes (required analytes spiked into QC samples) in standard matrices, to verify performance.

- Spikes of surrogates in all field samples, to observe recovery effects in the environmental matrix (if possible for the method).

Class 1P Methods:

- Method Blank, to verify that the laboratory is not a source of sample contamination; and

- Spikes of all control analytes (required analytes spiked into QC samples) in standard matrices, to verify performance.

- Spikes of surrogates in all field samples, to observe recovery effects in the environmental matrix.

Class 1M Method (GC/MS Only):

- Method Blanks/Spikes, to verify that the laboratory is not a source of sample contamination (non-surrogates) and to verify performance (surrogates); and

- Spikes of all control analytes (surrogate only) in every field sample, to observe recovery effects in the environmental matrix.

Class 2 Method:

- Method Blank, to verify that the laboratory is not a source of sample contamination; and

- Spikes of all control analytes in standard matrices, to verify method performance and to distinguish between the response of this sample and the response obtained from the blank.

Table 8-1. NUMBERS AND CONCENTRATIONS OF QC SAMPLES PER LOT

CLASS 1

1 - Standard Matrix Method Blank
3 - Standard Matrix Spikes
2 X MDL, 80% URL, 80% URL(approx)
All Field Samples - Natural Matrix Spikes
80% URL (approx.) Surrogates Only (if possible for the method)

CLASS 1P

1 - Standard Matrix Method Blank
4 - Standard Matrix Spikes
2 X MDL, 2 X MDL, 80% URL, 80% URL (approx.)
All field samples - Natural Matrix Spikes
80% URL (approx.) Surrogates Only

CLASS 1M

2 - Standard Matrix Method Blanks/Spikes
80% URL (approx.) Surrogates
All Field Samples - Natural Matrix Spikes
80% URL (approx.) Surrogates Only

CLASS 2

1 - Standard Matrix Method Blank
1 - Standard Matrix Spike
1 X MDL

NOTE 1: Wherever a spike level of 80% URL is specified this shall not exceed 20 X MDL.

NOTE 2: When a standard surrogate spiking level is specified in the method that level shall be used.

TABLE 8.2 CLASS 1P QC Spike Run Order

Number of 12 Hr. Periods	Period 1	Period 2	Period 3	Period 4
4	low	high	low	high
3	low	high high	low	---
2	low high	low high	---	---
1	all	---	---	---

8.2.2 PREPARATION OF CONTROL SAMPLES

Because QC samples are used for rapid, daily control of the analytical process, most QC samples must be identifiable by the analyst. Sample numbers for QC samples must be assigned during the logging-in and lot make-up process. However, actual preparation of the QC samples shall be performed by the person who conducts the first step of the analytical method. This person is responsible for obtaining the correct volume/weight and type of standard matrix (Sections 6.5 and 6.6) or field sample, and for spiking the matrix with the required analytes at the correct concentration (Section 8.2.1).

The spiking solvents and procedures will be specified in the approved method write-up. In general, however, the correct volume or weight of standard matrix/field sample for each method will be spiked with all control analytes using a minimum of spiking solution to prevent altering the character of the matrix. Spiked samples, excluding water samples and VOAs in soil, must be allowed to stand for one hour before continuing the analysis.

Validation of spiking solutions must be performed on a regular basis before the solution is used and not afterwards as part of a correction action. The following procedure shall be used:

Dilute working solutions will be validated against working standards before initial use and within seven days before subsequent usage. The method of validation should utilize the same technology used for measurement in environmental samples. GC/FID may be substituted for GC/MS with approval from the USAEC Chemistry Branch.

For single analyte solutions and the multi-analyte solutions used for other than GC/MS procedures, recovery must be greater than the lower warning limit in the control chart for that analyte. The control chart for the concentration closest to the solution concentration shall be used. If the same solution is used to spike water and soil, the control chart that exhibits the more stringent control limit shall be used. If a solution is suspected of deterioration at other times, it shall be tested before it is discarded to assess its status and allow judgements on spiked control samples prepared since the last solution validation.

For multi-analyte surrogate solutions for GC/MS, recovery of all surrogates shall be greater than the lower control limits on the X charts if GC/MS is used for validation. If GC/FID is used, the recovery shall be greater than the lower warning limit.

8.3 FIELD QC SAMPLES

Samples such as field blanks, trip blanks, rinse blanks, and field duplicates are collected by individuals performing sampling or contamination assessment. They must be specified when planning field activities and explicitly described in the Project Workplan. These samples should each be included at the rates indicated in Table 8-3. This table represents the general EPA requirements, and may be modified to meet site specific criteria, or additional regulatory requirements. Such field samples are not part of laboratory QC and will be treated by the laboratory simply as environmental samples. Evaluation of data from these field samples must be performed by the contractor when the final report is produced.

TABLE 8-3 FIELD QC SAMPLES

TYPE	FREQUENCY
Trip Blanks (VOAs in water only, unless required by regulatory agency)	1 per cooler shipped
Rinse Blanks (Not required for dedicated sampling equipment)	1 per day per equipment type
Field Blanks	1 per 20 samples
Field Duplicates	1 per 20 samples

Note that this table does not include the matrix spikes and matrix spike duplicates required by the EPA. The EPA uses these samples to determine matrix effects and within day variability of the laboratory. In lieu of these USAEC will perform the following:

- Matrix effects shall be determined using surrogates in each field sample, if appropriate surrogates are available. If surrogates are not available, then matrix spikes will be performed at a rate of 1 per 20 samples.

- The within day variability of the method will be determined using the USAEC required duplicate standard matrix QC spikes. This will replace the matrix spike duplicates. The use of a standard matrix ensures that any variability is done to laboratory performance, and not a result of matrix effect.

8.4 QA SPLIT SAMPLES

In addition to the QC samples listed above, the contractor (if so directed in the task order) shall provide 10 % split samples for analysis at a Corp of Engineers (COE) QA laboratory. The contractor is responsible for providing sample containers and coolers, and for the shipping of these samples to the QA laboratory. USAEC will advise the contractor where the samples are to be sent, and if any analyses are not to be split

(ie. those for which no COE laboratory has the capability to analyze for). USAEC will review the results from the splits and use this information as part of the overall data validation program.

Water samples and samples for volatiles shall be discrete, collocated samples. For all other parameters in soil the sample shall be thoroughly mixed prior to bottling of the fractions.

8.5 DATA REPORTING for QC

8.5.1 CLASS 1, CLASS 1P, and CLASS 1M METHODS

The results for each analyte in the spiked QC sample shall be determined using the same acceptable calibration curve that is used for environmental samples in the lot. Data shall be reported as "less than" the MDL if the analyte is not detected. Any values above the MDL shall be reported as determined. Values above the instrumental detection level (IDL), but below the MDL, shall be reported as determined, but must be flagged with "J" and "P" to indicate that the value is estimated. Results for QC samples shall not be corrected, except as described below. Because all spike levels must be within the reporting range, no dilutions should be required. Data shall be reported in the USAEC IRDMIS, as described in Section 9.6, using the correct number of significant figures (maximum of 3 for Class 1 and Class 1P, 2 for Class 1M and Class 2).

Each day of analysis, the analyst shall quantify each analyte in the method blank and spiked QC samples. A new lot of samples shall not be introduced into the analytical instrument until results for QC samples in the previous lot have been calculated, plotted on control charts as necessary, and the entire analytical method shown to be in control. If time is a constraint, the calculation of associated environmental sample results may be postponed until a later date. The analyst should maintain control charts by the instrument so that the results of QC samples could be hand-plotted, in order to have an early indication of problems.

Data from the method blank shall be reported as "less than" the MDL if the analyte is not detected. Any values above the MDL shall be reported as determined. Values above the instrumental detection level (IDL), but below the MDL, shall be reported as determined, but must be flagged with "J" and "P" to indicate that the value is

estimated. Corrections to the QC samples is required whenever an analyte is detected above the IDL in the method blank. The correction will be done based upon the instrument response values and not the found values calculated from a calibration curve. If the instrument response output is only available in concentration than this may be used. Entries into the USAEC IRDMIS shall be in terms of concentration. The importance attached to finding measurable concentrations in the method blank is dependent on analyte and method. In the Project QC Plan, each laboratory must describe its procedure for assessing method blank results and identifying laboratory contamination problems.

8.5.2 CLASS 2 METHODS

Method blank and dilution corrections are not performed for Class 2 analyses. The results for samples analyzed by Class 2 methods are measured in relation to the MDL (two significant figures) and reported as "less than, equal to, or greater than" the MDL. A tested concentration range is not applicable since only the MDL concentration is tested.

8.6 CONTROL CHARTS

Control charts are not used with Class 2 methods. For Class 1, Class 1P, and Class 1M methods, control charts are used to monitor the variations in the precision and accuracy of routine analyses and detect trends in these variations. The construction of a control chart requires initial data to establish the mean and range of measurements. The QC control charts are constructed from data representing performance of the complete analytical method.

Although tabulations of the various statistical parameters can be used to evaluate if a datum falls within the prescribed limits, trends are very difficult to discern from tables. Therefore, control charts shall consist of tabulated data and graphical portrayals of the information described below. Software packages that to be used to construct charts will be provided by USAEC and the use of the USAEC supplied software is required.

In the initial construction of the control charts, data from the laboratory analyses will be used. Data from spiked QC samples within a lot will be compared to control chart limits to demonstrate that analyses of the lot are under control, and will be used to update the charts. $\bar{x}$ - R control charts will be used in these guidelines.

Each control chart shall include the following information:

- Analyte;
- Method number;
- Laboratory;
- Spike concentration;
- Matrix; and
- Chart title - select one of the following:

  1) Single Day X-Bar Control Chart - High Spike Concentration

  2) Single Day X-Bar Control Chart - Low Spike Concentration

  3) Single Day Range Control Chart - High Spike concentration

  4) Single Day Range Control Chart - Low Spike concentration

  5) Three-Day X-Bar Control Chart - Low Spike Concentration

  6) Three-Day Range Control Chart - Low Spike Concentration

- Four letter lot designation for each point, shown on the x-axis;
- Percent Recovery (for $\bar{x}$ control charts) or Range (for R control charts) along the y-axis;
- Upper control limit (UCL), on $\bar{x}$ and R control charts;
- Upper warning limit (UWL)*, on $\bar{x}$* and R control charts;

- Mean, on $\bar{x}$ and R control charts;
- Lower warning limit (LWL)*, on $\bar{x}$* control charts; and
- Lower control limit (LCL), on $\bar{x}$ control charts.

* For some analytes specified by USAEC, warning limits on $\bar{x}$ charts will be deleted and replaced by modified control limits based on data quality specifications. See Appendix L for details.

If the method is judged to be out-of-control (Section 8.7) and reanalysis occurs, no point from the initial analysis may be used to update charts.

Specifics on the construction of control charts can be found in Appendix H.

8.7 OUT-OF-CONTROL SITUATIONS

Failure to meet calibration criteria, record keeping omissions, improper sampling technique, and improper storage or preservation of samples are all conditions that affect data quality and require investigation/correction. However, this section of the guidelines describes only evaluations performed by the analyst, in consultation with the QAC, to determine whether the entire analytical method is in control. These evaluations must be done daily so that action can be taken immediately to investigate and correct the problem. Failure to take immediate action may necessitate discarding large quantities of data and reacquiring, preparing, and reanalyzing samples processed after the problem was detected.

For both duplicate spiked QC results and moving averages a single mean (X) outside of modified limits requires immediate investigation/corrective action. When two or more successive lot means for duplicate spiked QC data are outside normal control limits but within modified limits, investigation/corrective action should be taken even though the data from these lots are acceptable. For moving averages, a single point outside of normal control limits but within modified limits requires investigation/corrective action even though the data are acceptable.

8.7.1 HOLDING TIMES

Any sample or sample extract held beyond the time periods specified in Section 6.5 shall be deemed out-of-control. These samples should not be analyzed unless incident-specific exception is received from USAEC. Sampling and laboratory schedules, and budgets, should be coordinated to avoid holding time violations.

8.7.2 $\bar{x}$ Control Charts

An out-of-control situation for $\bar{x}$ control charts may be indicated by:

- A value outside the control limits or classified as outlier by statistical test;
- A series of seven successive points on the same side of the central line;
- A series of five successive points going in the same direction;
- A cyclical pattern of control values; or
- Two consecutive points between the UWL and UCL or the LWL and LCL.

Note that for moving average control charts it is the individual daily recovery, not the average which must be evaluated.

Whenever one of these conditions is detected, the analyst and QAC must investigate to determine the cause and document actions taken. Data acquired concurrently with one of these conditions shall be discarded and samples reanalyzed unless the investigation of the problem proves that the analysis was in control, or modified control limits are being used to determine acceptability of data (See Appendix L). Justification for the acceptance of data must be provided with the weekly quality control submission.

The analyst will determine whether all sample analyses by a multi-analyte method should cease, in the following way:

- Plot average percent recovery ($\bar{x}$) for each analyte.

• If the points for at least two thirds (see Table 8-4) of the control analytes for a multi-analyte method are classified as in-control, based on the conditions described above, the method is in control and environmental sample data may be reported (providing that the condition of two consecutive out-of-control points has not occurred). The conditions which may have caused more than one third of the control analytes to fail the control criteria shall be investigated and corrected as necessary. All activities shall be documented. The data points indicating possible error shall be annotated with a reference to the investigation and to the fact that the method met control criteria.

• A method may be deemed out-of-control even if greater than or equal to 2/3 of the control analytes meet control criteria. Of the remaining control analytes (less than 1/3 possible out-of-control), if one analyte has two consecutive out-of-control points, as defined above, the method is out-of-control. Analyses must cease, the cause must be investigated and corrected, and a determination made by the USAEC Chemistry Branch of whether the lot must be reanalyzed.

• If data points for fewer than 2/3 of the control analytes are classified as in control (more than 1/3 meet one of the out-of-control conditions), the method is considered to be out-of-control and all work on that method (including sample preparation) must cease immediately. No data for environmental samples in that lot may be reported. Efforts must be initiated to determine the cause of the problem. If the problem is instrumental or specific only to preparation of that lot, samples prepared after the out-of-control situation occurred may be processed after the instrumental system is repaired and recalibrated, provided holding times are not exceeded. If no specific cause can be assigned, the instrument should be recalibrated and all samples prepared subsequent to the last in-control lot should be re-prepared, provided that the holding time has not expired. If the holding time has expired then USAEC must be contacted for guidance on re-sampling. In any case, the out-of-control lot shall be reanalyzed. The out-of-control situation and corrective actions taken shall be fully documented. Each point shall be annotated with a reference to the investigation and to the disposition of samples and results.

• The establishment of overall method control for analyses may not be accurate for describing a particular analyte(s). For analyses where control cannot be established for certain control analytes (i.e., loss of surrogate due to volatility), such analyte results may still be deemed as out-of-control even though the method is considered in control. The evaluation of control in such instances will be handled on a case-by-case basis.

If a lot is still out of control after reanalysis, all method-related activities shall stop immediately. A detailed laboratory-wide investigation shall be conducted to isolate and correct faulty operations. Sample security, integrity of standards, reagents, glassware, laboratory notebooks, instrument performance, and adherence to validated methods should be included in the investigation and the findings/corrective actions documented.

8.7.3 R CONTROL CHARTS

An out-of-control situation for R control charts may be indicated by:

- A value above the UCL;
- A series of five consecutive points going in an upward direction;
- A cyclical pattern of control values; or
- Two consecutive points between the UWL and UCL.

Whenever one of the conditions is detected, the analyst and QAC must investigate. Criteria for determining if a method is in control are the same as those described in Section 8.7. Out-of-control on range charts bears as much weight as out-of-control on accuracy charts.

Table 8-4. MINIMUM NUMBER OF IN-CONTROL POINTS FOR MULTI-ANALYTE METHODS

Required Control Analytes Per Method	Required Number of Data Values Falling Between the UCL and LCL
1	1
2	2
3	2
4	3
5	4
6	4
7	5
8	6
9	6
10	7
11	8
12	8
13	9
14	10
15	10
16	11
17	12
18	12
19	13
20	14
21	14
22	15
23	16
24	16
25	17

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9.0 DATA REDUCTION, VALIDATION, AND REPORTING

Traditionally, record keeping was the primary emphasis of QA. Although the primary emphasis of this USAEC QA Program is the control of sample analysis, record keeping maintains its importance in the overall assessment of the production of quality data and is used in part to document the control of sample analysis.

The degree of rigor used in documenting sampling and analysis activities cannot be understated. All activities require extensive documentation and special handling protocols. All activities are to be performed under chain of custody procedures. Particularly in these situations, the attitude is "if you didn't write it down, you didn't do it."

For most USAEC projects, this degree of documentation is required. However, for some projects, documentation in the form of an EPA CLP package will be required. In any case, the records described below shall be maintained and will be available for inspection by USAEC.

Note that the Daily QC Report requirement of ER 1110-1-263 is replaced by the USAEC requirement to maintain daily field logbooks during sampling activities. Copies of these logbooks will be submitted in lieu of the Daily QC Reports.

9.1 RECORD KEEPING

Bound logbooks with pre-numbered pages shall be utilized for record keeping. In addition to the pre-numbered pages, each logbook or laboratory notebook shall have a unique number for ease of identification. Additional documentation, such as chromatograms, shall be referenced to the logbook or notebook, where appropriate. Loose sheets are not to be used unless permanently affixed to the logbook. The use of bound books tends to result in a chronological sequence of data insertion. Numbered pages encourages use of data in sequence and also aids in referencing data through a table of contents ordered according to time, type of analysis, type of sample, and/or identity of analyst.

Validation can be easily accomplished by requiring the sampler or analyst to date

and sign each activity or analysis prior to the end of their work shift. This validation should be further strengthened by providing space for the supervisor to witness the date and the completion of the analyses.

Logbook entries shall be completed in ink. Corrections should be made by drawing one line through the incorrect entry, entering the correct information, initialling, and dating the change. Complete information should be entered so that in an examination it can be determined what was done, by whom, when, and what the results were. At the end of each work shift, the analyst shall sign after the last entry is made.

Computerized logging systems may be used as support tools during any record keeping activities. However, bound logbooks are required for original records. If computers are used, bound logbooks must nevertheless be maintained. A computer hardcopy that has been permanently affixed in the logbook is acceptable as an original record of sampling and laboratory logging.

Separate installation logbooks or partial logbooks in other formats (e.g., analytical lot) maintained in conjunction with the installation logbook are the preferred methods for documenting appropriate information relevant to chemical analyses performed during USAEC projects. Master instrument logbooks are acceptable; however, such logbooks generally remain permanent property of the laboratory. Whatever logbook practice is utilized should minimize the duplication of records and be identified in the project QC plan submitted to USAEC. Logging, tuning, calibration, and reporting activities must be included in the logbooks. Copies of laboratory notebooks that integrate non-USAEC projects shall not be acceptable. Routine maintenance activities (Chapter 6) do not require installation-specific logbooks.

At the end of a project, all logbooks containing information specific to the installation shall be forwarded to USAEC for maintenance. Corporate controlled logbooks should be avoided; however, if such logbooks are used by the laboratory; certified copies of all relevant logbook pages shall be submitted to USAEC. A certified copy is a copy with the source documented, signed, and dated, after copying, by the Laboratory Task Manager or Quality Assurance Coordinator.

Because exact procedures vary between laboratories, an exact system for documentation will not be specified. However, the records described in the following sections must be maintained for each USAEC project.

9.2 LABORATORY

9.2.1 LABORATORY LOGGING

Upon arrival at the laboratory, samples shall be logged into a bound laboratory book, preferably installation specific. Logging the samples into a laboratory-wide sample tracking system (logbook or computer) does not supplant the need for a written project-specific log. Sample information provided in the logbook must include:

- Field sample number;
- Date of arrival at the laboratory;
- Observations concerning the conditions under which the samples arrived, e.g., broken containers, leakage, temperature of cooler upon receipt, unusual appearance of samples, etc;
- Analyses requested; and
- USAEC sample identification number (in addition to any internal laboratory sample numbers) associated with each field sample number. The USAEC sample identification numbers must be sequential, including laboratory QC samples, in the format described in Section 6.4.
- When problems are encountered with samples the sampling contractor shall be notified. Written records shall be maintained of all communications with the sampling contractor.

Prior to the analysis, samples are grouped into analytical lots, ordered and assigned a USAEC sample identification number. The laboratory may use internal laboratory sample numbers in addition to the required USAEC designation. USAEC sample identification numbers will be assigned for the QC samples to ensure inclusion of the correct number of QC samples in each lot for each analytical method (Section 8.2.1).

9.3 ANALYTICAL RECORDS

Reference Materials:

A bound logbook record shall be maintained of all reference materials (Section 7.6) used on a project. The record shall include date of receipt, source, purity, all compositional information, storage conditions, and expiration date. Data obtained during characterization of purchased materials (Section 7.6.3) shall also be included.

Working standards made from reference materials shall be labeled with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date, and logbook where information on the standard is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations) shall also be recorded. There should be no bottle, flask, beaker, or vial that contains a sample, sample extract, or standard solution that is not correctly labeled and properly stored.

Sample Handling:

Each person conducting any part of an analytical protocol shall maintain a record of all activities in a bound logbook. This notebook shall be specific to the operation but need not be person-specific if several individuals perform the same operation. Each day the analyst shall record the samples handled, standards used, QC samples prepared, procedures used, and resultant calculations. The logbook shall be signed and dated daily.

9.4 DATA REPORTING

All numerical results shall be reported in terms of concentration in the environmental sample. Resultant found concentrations submitted for entry into the USAEC IRDMIS must remain unadjusted before being reported to USAEC. Correction factors (e.g., percent soil moisture and dilution factor) are maintained separately in the IRDMIS. All data must have been collected during periods when calibration and control systems were used. Data shall be reported as "less than" the MDL if the analyte is not detected. Any values above the MDL shall be reported as determined. Values above the instrumental detection level (IDL), but below the MDL, shall be reported as determined, but must be flagged with "J" and "P" to indicate that the value is estimated. Specific instructions are provided in the IRDMIS User's Guide regarding the coding of entries. Flagging codes, as described in the IRDMIS User's Guide will be used, when applicable, to comment on the data. Contractor Laboratory comments on the data are mandatory.

In reporting results, rounding to the correct number of significant figures should occur only after all calculations and manipulations at the laboratory are completed. As many figures as are warranted by the analytical technique should be used in pre-reporting calculations. Premature rounding can significantly affect the final result.

Rounding will be accomplished using the following rules:

Rule 1 - In expressing an experimental quantity, retain no digits beyond the second uncertain one.

Rule 2 - In rounding numbers (i.e., in dropping superfluous digits);

- Increase the last retained digit by one if the first uncertain digit is larger than 5;

- Retain the last digit unchanged if the first uncertain digit is less than 5; and

- Retain the last digit unchanged if even, or increase it by one if odd, if the first uncertain digit is 5 and the second uncertain digit is 0.

- Increase the last retained digit by one if the first uncertain digit is 5 and the second uncertain digit is >0.

The correct number of reported significant figures, by method class, are as follows:

- Class 1 and 1P - 3 significant figures;
- Class 1M - 2 significant figures; and
- Class 2 - 2 significant figures.

The number of allowable significant figures are reduced when added uncertainties are included in the analysis, i.e., the results for samples diluted into the Method Reporting Range (MRR) allow one less significant figure due to the uncertainty added by the dilution process.

When required by contract or task order, data may have to be reported according to EPA CLP format (as specified in the CLP Statement of Work), in addition to those described above.

9.4.1 CLASS 1, CLASS 1P, AND CLASS 1M METHODS

Class 1 and 1P Methods:

If results for an analyte were obtained using the method exactly as tested, without dilution, the analyte concentration in the sample may be reported to three significant figures. If dilution was required for a particular analyte, the result may be reported to only two significant figures.

Class 1M Methods:

Results for all analytes (target and surrogate) may be reported with two significant figures if the method was used without dilution. Results obtained after dilution and results of screening for non-target analytes may be reported to only one significant figure. Any results for Class 1M methods that result from manual integration of chromatographic peaks shall be justified with copies of the specific peaks (instrument integration and manual integrations) provided in the data package.

9.4.2 CLASS 2 METHODS

The results of Class 2 methods are not adjusted for dilution. The results for samples analyzed by Class 2 methods are measured in relation to the MDL (two significant figures) and reported as "less than, equal to, or greater than" the MDL. A tested concentration range is not applicable since only the MDL concentration is tested.

9.5 DATA DELIVERABLES

In addition to those requirements of providing the results of analyses, both for analytical samples and QC samples, to the USAEC Data Management System, the contractor laboratory is responsible for maintaining and providing to USAEC the following documentation:

- Data Package - A data package contains all the data necessary to support the results of one analytical method for one lot of samples. Data packages shall be "free standing," that is, all data should be available without reference to other documents or files. The data package shall be forwarded to USAEC at the completion of the project or as otherwise specified (i.e., delivery order package or case file package). The description of the contents of a data package and the requirement for their review are contained in Section 9.5.1. A data package is basically all back-up data for a CLP data package, without the CLP report forms. Therefore, it should be possible to produce a CLP report from the data contained in the data package, if required.

- Delivery Order Package - A delivery order package consists of all the data packages associated with a specific delivery order of a contract and will be forwarded to USAEC at the completion of the analyses specified in the delivery order.

- Case File - A case file consists of the data or data packages associated with a specific case as defined in the EPA Contractor Laboratory Program. When specified, data may be required to be delivered to USAEC following EPA CLP protocols (as defined in the CLP Statement of Work), at the completion of the analysis of a case lot of samples.

• Other - As required in a contract or delivery order, data and/or data packages may be required to be delivered to USAEC at a specified frequency other than those described above.

9.5.1 DEVELOPMENT AND USAGE OF DOCUMENT CONTROL PROCEDURES

9.5.1.1. PURPOSE AND DEFINITION

Document control procedures are necessary in order to produce a litigation quality data package. A data package shall contain all the data necessary to support the results of one analytical method for one lot of samples. Data packages shall be "free standing," that is all data shall be available without reference to other documents or files.

9.5.1.2 CONTENTS OF DATA PACKAGE

In general, all data shall be maintained in two separate locations, the data package and the laboratory notebook(s).

Records to be contained in the data package shall include, but are not limited to the following:

• Original chromatograms, strip charts, or other instrument output. Note that all run data must be included, even if it is not used in determining the final result.

• Original chain of custody form and carrier transmittal documents.

• All hardcopy GC/MS output.

• Expanded scale blow-up of manually integrated peak(s).

• All data sheets or other preprinted forms used by the contractor laboratory.

• All injection logs.

• One sample per lot shall have the ICP spectra printed in hard copy, if possible, according to the instrument used.

• Copies of all relevant notebook pages. This shall include preparation of calibration and QC spiking standards (from stocks to working standards), calibration, sample appearance, sample pH, sample preparation/extraction, moisture determinations, calculations, and any other relevant comments. When any preparation or analysis step is to be performed for a specified time (ie. sonicate for 18 hours) the start and stop times of the procedure shall be recorded.

• Corrective action and non-conformance reports.

• Hard copy of the transfer file as transmitted to USAEC.

Each data package shall contain all information related to one lot for one installation. In cases where a lot has samples from more than one installation then the information shall be copied and placed in separate packages for each installation. In those packages which receive copies, the location of the original material shall be identified.

Each data package shall contain a contents and approval checklist. This list shall identify all materials which must be placed into the data package. This list shall also list reviewer's names, dates of review, provide space for comments, notes, and corrective actions.

It is the responsibility of the contractor laboratory to review data packages for both content and correctness (see Section 9.5.1.3).

Included in the data package shall be a discussion of the observations of the data contained in that package. This discussion shall include, but not be limited to, observed matrix effects, unusual sample appearance, sample pH, blank results, control problems, deviations from approved SOPs, digressions from normal practices (i.e., manual integrations) and reasons thereof, etc. The impact on the usability of the data shall be discussed. Explanations on the use of the applicable flagging codes shall be provided.

9.5.1.3 REVIEW OF DATA PACKAGES

All data packages shall be reviewed by the contractor laboratory. This review should be completed no later than 30 days after the sample analyses for the lot are completed. This review serves two primary purposes. First it ensures that all required data and documents are contained in the data package. Secondly it checks the content for record keeping errors.

Reviewer's names and dates of review shall be recorded on the data package checklist. If any corrective actions are required they shall also be noted. When corrective actions are completed the reviewer shall place his/her initials and date next to the original comment to indicate completion of the action. The responsibility for final review of all data packages resides with the Quality Assurance Officer of the contractor laboratory. The final step in any evaluation shall be the attesting, in writing, of the Quality Assurance Coordinator as to the validity of the data.

Additional reviews are performed at USAEC after receipt of the data packages. Specific procedures for the reviews are covered in USAEC Chemistry Branch internal SOPs.

9.5.1.4 NOTEBOOKS

All contractor laboratories shall use bound notebooks. Both the sewn binding and the plastic binding (i.e., 19 ring GBC plastic binders) are acceptable. Pages shall be pre-numbered prior to use. Each notebook shall be assigned a unique notebook number which shall be recorded on the cover and on each page of the notebook.

Each page shall be signed and dated by the analyst and supervisor. Corrections shall be made by drawing a single line through the incorrect entry. Each correction shall be initialled and dated and also include a brief explanation for the correction. The use of correction media is prohibited.

If material is copied for inclusion in the notebook, the copy must be legible and not reduced to an excessive degree, making it unreadable.

9.5.1.5 FORMS

If the contractor laboratory uses preprinted forms for recording of data, then the original shall be placed into the data package and a copy retained in the appropriate notebook.

Forms should be designed to be specific to a given analysis. All spaces shall be filled, either with the required data or with an N/A to signify that the item is "not applicable" to the analysis.

Corrections shall be made with a single line through the incorrect entry, initialled, dated, and with a short explanation. The use of correction media is prohibited.

9.6 DATA MANAGEMENT SYSTEM

The results for samples analyzed in support of USAEC projects shall be entered in the USAEC IRDMIS. Specific instructions for format, coding, and submission are provided in the IRDMIS User's Guide. In order to facilitate correct and efficient data submission, the information listed in the IRDMIS User's Guide should be collected, recorded, and provided to contractor data management personnel. Questions pertaining to data management should be referred to the contractor data management group. Laboratories are encouraged to interface their internal data management system (i.e., LIMS) to the IRDMIS. USAEC will provide assistance in the accomplishment of that interface. A typical sequence of Data Management activities are shown in Figure 9-1. Any problems with USAEC provided software shall immediately be reported to the USAEC Chemistry Branch. Direct contact with the Data Management Contractor is discouraged, without prior notification of USAEC Chemistry Branch. When problems are site specific and may impact project performance Chemistry Branch will notify the COR/project officer.

Laboratories shall perform group and record checks of the data before transmission to the USAEC IRDMIS. However, the prime contractor is responsible for the quality and correctness of all data. Therefore, it is recommended that the prime contractor also group and record check the data. Any errors that can be corrected by the laboratory shall be corrected before transmission; otherwise the data will be returned

unprocessed. Data that cannot be corrected by the laboratory, e.g., results outside the MRR, will be reviewed by the USAEC Chemistry Branch for acceptance into the IRDMIS.

9.7 DATA REVIEW AND VALIDATION

An integral part of any QA Program is the review of data and its subsequent validation. The primary responsibility for this review and validation rests with the laboratory performing the analyses. Each data package must be reviewed with the data being validated prior to its submission to the Data Management System. Checklists, such as the examples in Appendix P, will be used to demonstrate that the data review was accomplished.

The data review and validation at the laboratory should include, but not be limited to, the following subjects:

- Completeness of laboratory data.
- Evaluation of data with respect to reporting limits.
- Evaluation of data with respect to control limits.
- Review of holding time data.
- Correlation of laboratory data from related laboratory tests.

The specific item for data review are covered in the Data Package Review Checklists, Appendix P.

Specific items for validation shall include, but are not limited to, the following:

- Examination of chain-of-custody records to ensure that custody was properly maintained.
- Comparison of data on instrument print-outs with data recorded on worksheets or in notebooks.

- Checking to ensure that the same calibration was used for all samples within a lot.

- Examination of chromatographic outputs and documentation of the reasons for manual integrations.

- Comparison of standard and sample preparation and injection records with instrument output to ensure that each output is associated with the correct sample.

- Examination of calibration and tuning results, to ensure that requirements are met.

- Checking calculations on selected samples to ensure correctness.

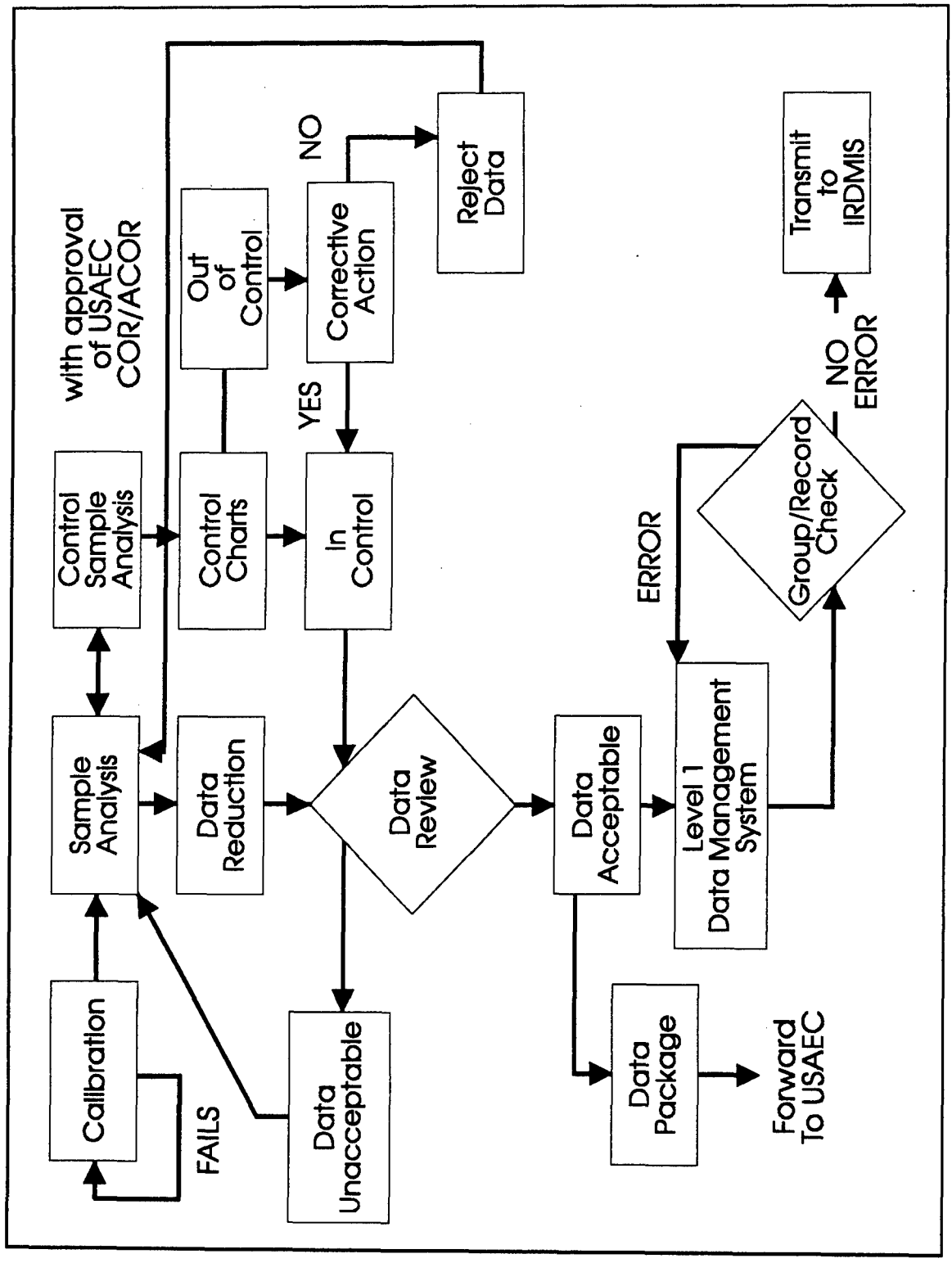
- Checking that GC/MS library searches have been performed for all unknowns, as required, and that the results have been evaluated and recorded.

- Examination of all papers and notebooks to ensure that all pages are initialed, dated, and have sufficient explanation for the changes, and that all items are legible.

- Comparison of transfer file, record and group check results with analysis results.

Similar reviews are performed at USAEC once the data packages are received.

Figure 9-1.


with approval of USAEC COR/ACOR
Out of Control
NO
Corrective Action
Reject Data
Transmit to IRDMS
Control Sample Analysis
Control Charts
YES
In Control
NO ERROR
ERROR
Group/Record Check
Sample Analysis
Data Reduction
Data Review
Data Acceptable
Level 1 Data Management System
Calibration
FAILS
Data Unacceptable
Data Package
Forward To USAEC

10.0 CORRECTIVE ACTIONS

When, as a result of audits or QC sample analysis, sampling or analysis systems are shown to be unsatisfactory, a corrective action shall be implemented. The Project Manager, Analytical Task Manager, QAC, and analyst shall be involved in the corrective action. If previously reported data are affected by the situation requiring correction or if the corrective action will impact the project budget or schedule, the action shall directly involve the Project Manager and the USAEC Project Officer and Project Chemist. Corrective actions are of two kinds:

- Immediate, to correct or repair nonconforming equipment and systems. The need for such an action will most frequently be identified by the analyst as a result of calibration checks and QC sample analyses.

- Long term, to eliminate causes of nonconformance. The need for such actions will probably be identified by audits. Examples of this type of action include:

  - Staff training in technical skills or in implementing these guidelines;

  - Rescheduling of laboratory routine to ensure analysis within allowed holding times;

  - Identifying vendors to supply reagents of sufficient purity; and

  - Revision of Contractor QA system or replacement of personnel.

For either immediate or long-term corrective actions, steps comprising a closed-loop corrective action system are as follows:

- Define the problem;

- Assign responsibility for investigating the problem;

- Investigate and determine the cause of the problem;

- Determine a corrective action to eliminate the problem;

- Assign and accept responsibility for implementing the corrective action;
- Establish effectiveness of the corrective action and implement the correction; and
- Verify that the corrective action has eliminated the problem.

The occurrence of the problem, corrective action employed, and verification that the problem has been eliminated must be documented.

In addition, if the corrective action results in the preparation of a new standard or calibration solution(s), then a comparison of the new versus the old solution needs to be performed and the results supplied with the weekly QC submittal as verification that the problem has been eliminated.

11.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Normal submissions to USAEC shall include the IRDMIS submissions (Section 9.6), audit reports (Section 12.0), and the results of QC activities (Section 8.0). When required in the task order, a CLP data package (as defined in the CLP Statement of Work) shall also be submitted. During those periods when analyses are being conducted, all QC charts (tabular and graphical), as described in Section 8.6, shall be submitted to the USAEC Chemistry Branch on a weekly basis. The QC report shall be provided to the Chemistry Branch NLT 5 working days after analyses for a week are completed. Analysis date shall be defined by the day the analytical instrument was run (Section 9.3). All points which indicate an out-of-control situation shall be evaluated and explained. Any corrective measures and reanalysis of samples shall be fully explained and documented, including procedural changes to prevent recurrence. Printouts generated from control chart software programs provided by USAEC shall be utilized, when available. A checklist for inclusion with each control chart submission is shown in Appendix M. In addition, for the first lot analyzed for each method, a copy of the calibration curve used for that lot shall be included.

As an appendix to the project final report, the QAC, in coordination with the Analytical Task Manager and the Project Manager, shall provide tabulation of all QC sample data, as well as specific observations delineating the control effectiveness for each analytical method. These observations will include the following:

- QC samples in each lot and how analytical results were combined to prepare control charts;

- Spike levels and rationale for choosing those levels;

- Possible effects on environmental sample results of detected concentrations in method blanks; and

- Unique matrix characteristics of environmental samples.

If at any time during the analytical effort a process was not in control, a discussion will be submitted on:

- Rationale for judging a point as in control, if it appears to satisfy an out-of-control

criterion listed in Section 8.7;

- Investigation of the out-of-control situation;
- Actions taken to bring the process back into control;
- Actions taken to ensure that the out-of-control situation did not recur; and
- Disposition of data acquired while the process was out-of-control.

12.0 PERFORMANCE AND SYSTEM AUDITS

An audit is a systematic evaluation to determine the quality of operation of some system or function. As applied in these guidelines, an audit may be external or internal.

12.1 EXTERNAL

External audits are conducted by representatives of the USAEC Chemistry Branch and prime contractors. These audits may be simultaneous or separate. After reviewing the proposed Project QC Plan, the Contractor Laboratory may be visited to discuss any weaknesses in the plan and to evaluate the laboratory's capability to implement the plan. During this visit, the USAEC representative may fill out the Audit Checklist (Appendix Q). Copies of the audit report will be provided to the USAEC Project Officer, the Contractor Project Manager, the Contractor Analytical Task Manager, the Contractor QAC, and the USAEC Chemistry Branch. If deficiencies are of a serious nature, copies will be forwarded to the Contracting Officer at Procurement for official documentation and action. The visit may occur before analyses of field samples are initiated by the laboratory.

After initiation of the analyses by the Contractor Laboratory, a USAEC representative may visit the field activities or the laboratory to evaluate the effective implementation of the Project QC Plan. Any project related activities may be evaluated during the visit (Appendix Q). Any documents or data required by the QA Program shall be eligible for inspection. Any aspect of the internal audit (as described in section 12.2) may be monitored. Findings will be reported to the USAEC Project Officer, the Contractor Project Manager, the Contractor Analytical Task Manager, the Contractor QAC, and the USAEC Chemistry Branch. If deficiencies are of a serious nature, copies may be forwarded to the Contracting Officer at Procurement for official documentation and action.

Scheduling/completion of the visits noted above does not preclude additional visits, as deemed necessary or desirable.

12.2 INTERNAL

Internal audits shall be conducted by the project QC staff (QAC or representative of the QAC) and shall include:

- Verification that standards, procedures, records, charts, magnetic tapes, etc., are properly maintained;

- Verification that actual practice agrees with written instructions; accomplished through the use of a systems audit where a selected method is monitored through all the steps of its performance. This system audit must be accomplished at least once each quarter, if the laboratory effort is long term; or once a month if the laboratory effort is short term. Methods must be selected so that all phases of a laboratory's effort is monitored, to include but not be limited to sample logging, chain of custody, sample preparation, standard preparation, extract storage and analysis and data reduction;

- Verification that QA records are adequately filed and maintained so as to assure protection and retrievability; and

- Assessment of results of QC sample analyses.

Auditing shall consist of observations and notations as to whether approved practices are followed. A formal audit report comprised of summary findings shall be distributed to the Project Manager, Analytical Task Leader, and USAEC Chemistry Branch. Deviations shall be noted and discussed with the staff member, appropriate management, and with USAEC. The audit and findings, both compliance and non-compliance, shall be documented in a bound logbook, or permanently attached and maintained as part of the QA documentation. The QA office shall maintain by project, a file(s) of audit reports and findings, copies of reports and findings that cover more than one project shall be maintained in each project file. At the conclusion of a project or task order, copies of the QA file shall be transmitted to the USAEC Chemistry Branch, along with the data packages.

12.3 FREQUENCY

Internal audits shall be conducted at least quarterly, and the results reported to USAEC within 2 weeks. Prime contractors shall conduct at least one laboratory audit per sampling event, or semiannually, whichever is greater. A written report of the audit shall be provide to USAEC within two weeks.

The USAEC will conduct audits at a frequency commensurate with the needs of the program/project.

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14.0 GLOSSARY

Accuracy -- Difference between individual analytical measurements and the true value, corresponding to the sum of systematic and random errors.

Analyte -- Chemical component for which analysis is conducted.

Analytical Method -- Set of written instructions completely defining the procedure to be adopted by the analyst in order to obtain an analytical result.

Audit -- Systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: 1) performance audits in which quantitative data are independently obtained for comparison with routinely obtained data in a measurement system; or 2) system audits of a qualitative nature that consist of an onsite review of a laboratory's quality assurance system and physical facilities for sampling, calibration, and measurement.

Chain-of-Custody -- Formalized system of creating an accurate written record which can be used to trace the possession and handling of a sample from the moment of collection through analysis and introduction of data as evidence.

Chemical Calibration Curve -- Best-fit regression curve determined from a plot of response versus calibration standard concentration.

Chemical Calibration Standard -- Solutions containing known amounts of analytes, introduced directly into the instrument to obtain the response versus concentration relationship for each analyte.

Comparability -- Confidence with which one data set can be compared to another.

Confidence Limit -- One of the end points of an interval which has a specified probability of containing a given parameter or characteristic.

Contractor Laboratory -- Analytical chemistry laboratory performing analysis of environmental samples in support of a USAEC contract. The laboratory may be part of the organization holding the contract with USAEC (prime contractor) or may be subcontracted to the prime contractor.

Control Analyte -- Analyte spiked into a QC sample. Control analytes may consist of target and/or surrogate analytes. Control charts are required for each control analyte.

Control Samples -- Samples introduced into the train of environmental samples as monitors on the performance of the analytical method (Section 8.2).

Data Package -- A data package contains all the data necessary to support the results of one analytical method for one lot of samples. Data packages shall be "free standing," that is, all data should be available without reference to other documents or files.

Data Validation -- Systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verification, and review.

Data Quality -- Totality of features and characteristics of a data set that bears on its ability to satisfy a given purpose.

Development Laboratory -- Laboratory designated and/or contracted to develop an analytical method.

Field Blank -- Standard matrix sample, to which no analyte of interest has been added, that is transported to the sampling site and back, to ensure that no contamination is introduced during shipment. This sample is created by pouring the distilled water used in the field into a randomly selected container at the sampling site.

Field Duplicate -- A second sample from one site taken in the field and submitted to the laboratory as a separate sample. It is usually analyzed "blind" by the laboratory, i.e., the laboratory does not know that it is a duplicate of another sample. The results act as an external check on the combined precision of sampling and analysis.

Found Concentration -- Concentration based on instrumental response of the sample compared to the instrument calibration curve.

Holding Time -- The maximum time allowable between sample collection and analysis.

IRDMIS -- Installation Restoration Data Management Information System, a USAEC computerized data submittal, storage, and retrieval system.

Lot Size, Maximum -- Number of samples, including QC samples, that can be processed through the rate limiting step of the analytical method during a single time period.

Method Blank -- Standard matrix sample to which no analyte of interest has been added that is processed in the same manner as samples, to ensure that the apparatus and reagents used are not contributing contaminants to the analysis.

Method Documentation Package -- A detailed description of the method to be performed.

Method Detection Level -- The lowest level at which an analyte may be reported.

Method Reporting Range -- The range of concentrations from which data may be reported. This is the range between the Method Detection Level and the Upper Reporting Level.

Negative Interference -- A response indicating a lesser amount of analyte than is actually present.

Outlier -- An extreme observation that is shown to have a low probability of belonging to a data population.

Percent Imprecision -- Single concentration standard deviation divided by the average found concentration; also called Relative Standard Deviation.

Percent Inaccuracy -- The difference between the found and target (true) concentration, divided by the target concentration and multiplied by 100.

Positive Interference -- A response indicating the presence of an analyte in greater amounts than actually present.

Precision -- Degree of mutual agreement among individual measurements made under prescribed conditions with a single test procedure.

Project QC Plan -- An orderly assembly of detailed and specific procedures which delineates how data of known and accepted quality are produced for a specific project.

Project Officer -- The individual responsible for the project at USAEC. The Project Officer may be the USAEC Chemistry Branch Chemist assigned to the project or the Contract COR, depending on the contract under which work is being performed.

Quality Assurance (QA) -- The total integrated program for assuring and documenting the reliability of monitoring and measurement data and for integrating quality planning, quality assessment, and quality improvement efforts to meet user requirements.

Quality Control (QC) -- The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

Quality Control Sample -- Sample that is introduced into a train of environmental samples as a monitor on the performance of the analytical system.

Rank of an Observation -- The number assigned to an observation if a collection of observations is ordered from smallest to largest and each observation is given the number corresponding to its place in the order.

Recovery -- Difference between the analytical results before and after spiking, divided by known amount of spiking compound and multiplied by 100 to convert to percentage.

Representativeness -- The degree to which data accurately and precisely represent a characteristic of a populations parameter variations at a sampling point, a process condition, or an environmental condition.

Response Factor -- The change in the size of peaks of standards that are run under the same conditions. The areas and retention time of the standards should not vary.

Rinse Blank -- Analyte free water which is poured over cleaned equipment and collected for analysis. The results are used to verify the efficiency of the equipment cleaning procedures.

Scientific Notation -- A method of expressing a number with the first significant digit to the left of the decimal point, the remaining significant digits to the right of the decimal point, and multiplied by ten raised to a positive or negative integer power.

Sensitivity -- Instrument response (counts, peak area, etc.) observed for the absolute quantity of analyte introduced into the instrument at the reporting limit.

Significant Figures -- The number of digits used to express a result in scientific notation. All digits are expected to be known definitely, except the last digit, which may be in doubt.

Spiked Sample -- A sample to which a known amount of analyte is added and which is then carried through the complete analytical method.

Standard Deviation -- The positive square root of the expected value of the square of the difference between a random variable and its mean.

Standard Sample -- Sample prepared in a standard matrix as defined in Sections 6.6 and 6.7.

Standing Operating Procedure (SOP) -- A written document which details an operation, analysis or action whose mechanisms are thoroughly prescribed and which is commonly accepted as the method for performing certain routine or repetitive tasks.

Target Analyte -- Specific, validated analyte reported for every sample analyzed by a given method.

Target Concentration -- Known spiked concentration.

Traceability -- The ability to completely reconstruct all activities from the time of sampling to data reporting, including all sample handling as well as instrument maintenance, QC results, and calibration curves.

Trip Blank -- A means to determine if volatile samples are being contaminated during shipping and storage. Vials of analyte free water are prepared by the laboratory and shipped to the sampling site and stored along with the empty sample containers. One trip blank shall be included in each cooler containing field samples for volatiles.

Upper Reporting Level -- The highest concentration at which an analyte may be reported, without the use of dilutions.

Validity -- Degree to which the reported results represent that which they intend to represent.

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15.0 LIST OF ACRONYMS

AAS -- Atomic Absorption Spectroscopy

ASTM -- American Society for Testing and Materials

BC -- Base Closure

BOD -- Biochemical Oxygen Demand

CLP -- Contract Laboratory Program

COD -- Chemical Oxygen Demand

EPA -- U.S. Environmental Protection Agency

GC -- Gas Chromatograph(y)

IC -- Ion Chromatograph(y)

ICP -- Inductively Coupled Plasma-Emission Spectroscopy

IDL -- Instrument(al) Detection Level

IR -- Installation Restoration

IRDMIS -- Installation Restoration Data Management Information System

IRM -- Interim Reference Material

LCL -- Lower Control Limit

LOF -- Lack of Fit

LWL -- Lower Warning Limit

MDL -- Method Detection Level

MRR -- Method Reporting Range

MS -- Mass Spectroscopy

NIST -- National Institute of Standards and Technology

NMR -- Nuclear Magnetic Resonance

QA -- Quality Assurance

QAC -- Quality Assurance Coordinator

QC -- Quality Control

RDL -- Required Detection Level

SARM -- Standard Analytical Reference Material

SRM -- Standard Reference Material from NIST

TDS -- Total Dissolved Solids

TOC -- Total Organic Carbon

TSS -- Total Suspended Solids

TRL -- Target Reporting Limit

UCL -- Upper Control Limit

URL -- Upper Reporting Level

USAEC -- U.S. Army Environmental Center (formerly known as the U.S. Army Toxic and Hazardous Materials Agency)

UWL -- Upper Warning Limit

ZI -- Zero Intercept

APPENDIX A

DOCUMENTATION FOR PROPOSED METHOD DEVELOPMENT

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APPENDIX A

DOCUMENTATION FOR PROPOSED METHOD DEVELOPMENT

1. Organization submitting documentation.

2. Statement of the problem.

3. Description of the technical approach to include specific details on procedures, solvents, instrumentation, etc.

4. Estimate of resources required to include labor hours, funds, and schedule.

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APPENDIX B

RANK SUM TEST

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APPENDIX B

RANK SUM TEST

The following pages contain examples of the Rank Sum Test used for evaluating Class 2 method performance data. The calculations are not performed by the computer software supplied by USAEC. The Rank Sum Test calculations shall be submitted as part of the Validation Procedure for Class 2 methods.

Table B-1. Rank Sum Test, Example 1

Standard Sample	Results	Rank	Average Rank
Blank	NN	1	2.5
Blank	NN	2	2.5
Blank	NN	3	2.5
Blank	NN	4	2.5
Spike	PP	5	6.5
Spike	PP	6	6.5
Spike	PP	7	6.5
Spike	PP	8	6.5

* NN = Negative; PP = Positive

** Average Rank for Negative Results = $\frac{1 + 2 + 3 + 4}{4} = 2.5$

Average Rank for Positive Results = $\frac{5 + 6 + 7 + 8}{4} = 6.5$

Sum of Average Ranks for Blanks = 2.5 + 2.5 + 2.5 + 2.5 = 10.

The criterion for acceptability is that the sum of the average ranks of blanks be less than or equal to 10. Therefore, the results are acceptable.

Table B-2. Rank Sum Test, Example 2

Standard Sample	Results*	Rank	Average Rank**
Blank	NN	1	2
Blank	NN	2	2
Blank	NN	3	2
Blank	PP	4	6
Spike	PP	5	6
Spike	PP	6	6
Spike	PP	7	6
Spike	PP	8	6

* NN = Negative; PP = Positive

** Average Rank for Negative Results = $\frac{1 + 2 + 3}{3} = 2$

Average Rank for Positive Results = $\frac{4 + 5 + 6 + 7 + 8}{5} = 6$

Sum of Average Ranks for Blanks = 2 + 2 + 2 + 6 = 12.

Because the sum of the average ranks of blanks exceed the criterion of less than or equal to 10, the results are unacceptable, therefore,

Test an additional two blanks and two spikes:

Standard Sample	Results	Rank	Average Rank ***
Blank	NN	1	3
Blank	NN	2	3
Blank	NN	3	3
Blank-New	NN	4	3
Blank-New	NN	5	3
Blank	PP	6	9
Spike	PP	7	9
Spike	PP	8	9
Spike	PP	9	9
Spike	PP	10	9
Spike-New	PP	11	9
Spike-New	PP	12	9

*** Average Rank for Negative Results = $\frac{1 + 2 + 3 + 4 + 5}{5} = 3$

Average Rank for Positive Results = $\frac{6 + 7 + 8 + 9 + 10 + 11 + 12}{7} = 9$

Sum of Average Ranks for Blanks = 3 + 3 + 3 + 3 + 3 + 9 = 24.

Because the sum of the average ranks of blanks meet the criterion of less than or equal to 26, the results are acceptable.

Table B-3. Rank Sum Test, Example 3

Standard Sample	Results*	Rank	Average Rank**
Blank	NN	1	3
Blank	NN	2	3
Blank	NN	3	3
Blank	NN	4	3
Spike	NN	5	3
Spike	PP	6	7
Spike	PP	7	7
Spike	PP	8	7

* NN = Negative; PP = Positive

** Average Rank for Negative Results = $\frac{1 + 2 + 3 + 4 + 5}{5} = 3$

Average Rank for Positive Results = $\frac{6 + 7 + 8}{3} = 7$

Sum of Average Ranks for Blanks = 3 + 3 + 3 + 3 = 12.

Because the sum of the average ranks of blanks exceed the criterion of less than or equal to 10, the results are unacceptable, therefore,

Test an additional two blanks and two spikes:

Standard Sample	Results	Rank	Average Rank ***
Blank	NN	1	3.5
Blank	NN	2	3.5
Blank	NN	3	3.5
Blank	NN	4	3.5
Spike	NN	5	3.5
Blank-New	NN	6	3.5
Blank-New	PP	7	9.5
Spike	PP	8	9.5
Spike	PP	9	9.5
Spike	PP	10	9.5
Spike-New	PP	11	9.5
Spike-New	PP	12	9.5

*** Average Rank for Negative Results = $\frac{1 + 2 + 3 + 4 + 5 + 6}{6} = 3.5$

Average Rank for Positive Results = $\frac{7 + 8 + 9 + 10 + 11 + 12}{6} = 9.5$

Sum of Average Ranks for Blanks = 3.5 + 3.5 + 3.5 + 3.5 + 3.5 + 9.5 = 27.

Because the sum of the average ranks of blanks exceed the criterion of less than or equal to 26, the results are unacceptable. The target concentration must be increased or a different method must be used.

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APPENDIX C

SAMPLE CONTAINER CLEANING PROCEDURES

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APPENDIX C

SAMPLE CONTAINER CLEANING PROCEDURES

To ensure the integrity of aqueous and solid samples, steps must be taken to minimize contamination from the containers in which they are stored. If the analyte(s) to be determined are organic in nature, the container should be made of amber glass. If the analyte(s) are inorganic, the container should be polyethylene. When both organic and inorganic substances are expected to be present, separate samples should be taken. New sample bottles must be cleaned according to either of the procedures presented below; reuse of sample containers is expressly prohibited. The procedure that was used must be documented. Commercially cleaned containers may be utilized if cleaning procedures comply with those provided in this appendix and prior USAEC Chemistry Branch approval is obtained. The procedures for cleaning the glass and polyethylene containers and their caps are as follows:

Specified by EPA for CLP

- Amber Glass Bottles

(1) Wash containers, closures, and teflon liners in hot tap water with laboratory grade non-phosphate detergent.

(2) Rinse three times with tap water.

(3) Rinse with 1:1 nitric acid.

(4) Rinse three times with ASTM Type 1 deionized water.

(5) Rinse with pesticide grade methylene chloride.

(6) Oven dry.

(7) Remove containers, closures, and teflon liners from oven.

(8) Place teflon liners in closures and place closures on containers. Attendant to wear gloves and containers not to be removed from preparation room until sealed.

- 40 mL Borosilicate Glass Vials

(1) Wash vials, septa, and closures in hot tap water with laboratory grade non-phosphate detergent.

(2) Rinse three times with tap water.

(3) Rinse three times with ASTM Type 1 deionized water.

(4) Oven dry vials, septa, and closures.

(5) Remove vials, septa, and closures from oven.

(6) Place septa in closures, teflon side down, and place on vials. Attendant to wear gloves and vials not to be removed from preparation room until sealed.

- High Density Polyethylene Bottles

(1) Wash bottles, closures, and teflon liners with hot tap water with laboratory grade non-phosphate detergent.

(2) Rinse three times with tap water.

(3) Rinse with 1:1 nitric acid.

(4) Rinse three times with ASTM Type 1 deionized water.

(5) Air dry in contaminant-free environment.

(6) Place liners in closures and place closures on bottles. Attendant to wear gloves and bottles not to be removed from preparation room until sealed.

Documentation must be provided to the USAEC Chemistry Branch validating that the bottles are in fact "clean." Documentation may consist of the results of "bottle

blank" analysis using the method(s) that will be applied to the sample that will be placed in that bottle. QC results from the supplier of commercially cleaned containers, demonstrating that the bottle(s) are "clean," will be acceptable. The documentation must be provided before the bottles are used to collect samples in the field. This validation is to be performed or provided for each batch or "lot" of bottles cleaned together and must be provided at least once for each installation where they are used.

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APPENDIX D

STANDING OPERATING PROCEDURES
FIELD OPERATIONS

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APPENDIX D

STANDING OPERATING PROCEDURES
FIELD OPERATIONS

The organization shall have written Standing Operating Procedures (SOPs) for all procedures and methods. SOPs shall be available for the following areas and shall contain, at a minimum, the information described.

- Training -- These SOPs describe the training procedures used to ensure that field personnel are qualified to perform the required functions.

- Sample Management -- These SOPs describe the numbering and labeling system, chain-of-custody procedures, and tracking of samples from collection to shipment or relinquishment to the laboratory. Sample management also includes the specification of holding times, volume of sample required by the laboratory, preservatives, and shipping requirements.

- Numbering and Labeling -- These SOPs describe the system for numbering and labeling samples. The numbering system shall ensure that a sample from a given location is assigned a unique number, and typically involves codes that explain information about the sample, such as matrix type, location, depth, and well number. The labeling SOPs shall specify the types of labels and markers to be used, typically waterproof, and the information to be included on the label, such as sample number, date and time of collection, sampler's name, matrix type, and type of analysis required.

- Sample Tracking -- These SOPs describe the procedures used to ensure that sample integrity is maintained from sampling and shipping through receipt in the laboratory. Chain-of-custody will be maintained and, therefore, possession shall be traceable from the time the samples are collected, through analysis, and finally to disposal. Typical information recorded on the custody form includes project name, signature of sampler(s), sampling station number, date and time of collection, and grab or composite sample designation. The signature of the individual(s) involved in sample transfer (i.e., relinquishing and accepting samples) must be documented.

- Sample Containers -- SOPs shall detail the specifications, including type and

size of container and lid, for each container used in a sample collection activity. In addition, SOPs shall specify cleaning procedures to be followed prior to the use of the container to ensure that the container does not contaminate the sample. SOPs may also specify protocols for verifying the cleanliness of the containers through chemical analysis.

- Sample Preservation and Storage -- Preservation techniques are generally limited to pH control, chemical addition, refrigeration, and freezing. SOPs shall describe which preservation techniques apply to a method, how preservatives are added, the amount added, procedures associated with shipping the preservative to the site, and any special handling or safety requirements.

- Holding Times -- Many analyses have a maximum time between collection and initiation of analytical work specified by either the method or regulations. If this time is exceeded, the analytes of interest may degrade and the data may be unusable. SOPs shall list holding times, if applicable, by method and sample matrix, and describe procedures for communicating holding time requirements to field personnel so that samples can be shipped to the laboratory in a timely manner.

- Shipping -- If the laboratory and sampling site are not in close proximity, the samples must be shipped. SOPs shall specify packaging procedures that prevent spills, maintain the required temperature, and meet Department of Transportation (DOT) requirements for shipping environmental or potentially hazardous samples. Instructions shall be provided for completing shipping papers. If holding times are crucial, SOPs should specify delivery to the laboratory within 24 hours or on weekends.

- Decontamination -- These SOPs describe the procedures used to clean field equipment before and during the sample collection process. The SOPs should include cleaning materials used, the order of washing and rinsing with the cleaning materials, requirements for protecting or covering cleaned equipment, procedures for disposing of cleaning materials, and safety considerations.

- Sample Collection Procedures -- SOPs for sample collection procedures shall describe how the procedures are actually performed in the field and not be a simple reference to standard methods, unless a procedure is performed exactly as described in the published method. The SOP for sample collection procedures should include the following:

- Applicability of the procedure;
- Equipment required;
- Detailed description of procedures to be followed in collecting the samples;
- Common problems encountered;
- Precautions to be taken; and
- Health and safety considerations.

It should include a statement that every effort shall be made to collect samples during the work week with samples delivered to the laboratory that same week.

- Corrective Action -- These SOPs describe procedures used to identify and correct deficiencies in the sample collection process. These should include specific steps to take in correcting deficiencies such as performing additional decontamination of equipment, resampling, and additional training of field personnel in methods procedures. The SOP shall specify that each corrective action must be documented with a description of the deficiency, the corrective action taken, and the person(s) responsible for implementing the corrective action.

- Records Management -- These SOPs describe the procedures for generating, controlling, and archiving field records. The SOPs should describe the responsibilities for record generation and control and the policies for record retention, including type, time, security, and retrieval and disposal authorities. Records shall include:

Project-specific records related to fieldwork performed for a group of samples. Project records may include correspondence, chain-of-custody, field notes, all reports issued as a result of the work, training records, project planning documents, and procedural SOPs used.

Field operations records, which document overall field operations. These records may include equipment performance and maintenance logs, personnel files, general field SOPs, and corrective action reports.

- Chemical and Sample Disposal -- These SOPs describe the policies and procedures for disposal of neat chemicals and standard and reagent solutions used in calibration of field equipment and decontamination procedures. Disposal of all chemicals must conform to federal, state, and local regulations.

- Reporting -- These SOPs describe the process for reporting the results of field

activities.

In addition, where analyses are performed in the field, the following additional SOPs are required:

- Reagent/Standard Preparation -- These SOPs describe the procedures used to prepare and document every standard and reagent solution used in field operations. Information concerning specific grades of materials used in the preparation, appropriate glassware, containers for preparation, storage, labeling, recordkeeping for stocks and dilutions, and safety precautions to be taken should be included.

- Equipment Calibration and Maintenance -- These SOPs describe procedures used to ensure that field equipment and instrumentation are in working order. The SOPs describe calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, service contractors or service arrangements for all equipment, and spare parts available in-house. Calibration and maintenance of field equipment and instrumentation shall be in accordance with manufacturers' specifications and shall be documented.

- Field Analysis -- All in situ, portable analysis, mobile labs, or other methods used in the field to determine a chemical or physical parameter shall be described by one or more SOPs. The SOPs shall incorporate applicable criteria from Appendix G.

- Data Reduction and Validation -- These SOPs describe procedures used to compute results from field measurements and to review and validate these data. They should include all formulas used to calculate results and procedures used to verify independently that field measurement results are correct.

APPENDIX E

CHAIN-OF-CUSTODY PROCEDURES

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APPENDIX E

CHAIN-OF-CUSTODY PROCEDURES

The material presented here briefly summarizes the major aspects of chain-of-custody. Reference should be made to NEIC Policies and Procedures (EPA-300/9-78-001-R) for more information.

E.1 INTRODUCTION

As in any other activity that may be used to support litigation, government agencies must be able to provide the chain-of-possession and custody of any samples which are offered for evidence or which form the basis of analytical test results introduced into evidence in any legal proceeding. It is imperative that written procedures be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed. The primary objective of these procedures is to create an accurate written record which can be used to trace the possession and handling of the sample from the moment of its collection through analysis and its introduction as evidence.

A sample is in someone's "custody" if:

- It is in one's actual physical possession;
- It is in one's view, after being in one's physical possession;
- It is in one's physical possession and then locked up so that no one can tamper with it; or
- It is kept in a secured area, restricted to authorized personnel only.

E.2 SURVEY PLANNING AND PREPARATION

The evidence-gathering portion of a survey should be characterized by the minimum number of samples required to give a fair representation of the sampled area or matrix. To the greatest extent possible, the number of samples and sampling locations should be determined prior to the survey.

All survey participants will receive a copy of the survey study plan and will be knowledgeable of its contents prior to the survey. A pre-survey briefing will be held to re-appraise all participants of the survey objectives, sample locations, and chain-of-custody procedures. After all chain-of-custody samples are collected, a debriefing will be held in the field to determine adherence to custody procedures and whether additional evidentiary samples are required.

E.3 SAMPLE COLLECTION, HANDLING, AND IDENTIFICATION

It is important that a minimum number of persons be involved in sample collection and handling. Standard field sampling techniques, such as those published by the U.S. Environmental Protection Agency, should be used for sample collection, preservation, and handling. Field records should be completed at the time the sample is collected and should be signed or initialed, including the date and time, by the sample collector(s). Field records should contain the following information:

- Unique sample or log number;
- Date and time;
- Source of sample (including name, location, and sample type);
- Preservative used;
- Analyses required;
- Name of collector(s);
- Pertinent field data (pH, temperature, depth to water, etc.); and

- Serial number of custody seals and transportation cases.

Each sample is identified by affixing a pressure sensitive gummed label or standardized tag on the container(s). This label should contain the sample identification number, date and time of sample collection, source of sample, preservative used, and the collector's initials. Analyses required should be identified. After all information has been recorded the label should be covered with water-proof tape. Where a label is not available, the same information should be affixed to the sample container with an indelible, water-proof marking pen.

The sample container should then be placed in a transportation case along with the chain-of-custody record form , pertinent field records, and analyses request form as needed. All records should be placed in a plastic, zip-lock type bag.The transportation case should then be sealed and labeled. All records should be filled out legibly in pen.

The use of the locked and sealed chests may never eliminate the need for close control of individual sample containers. Therefore, the sampler should place a custody seal around the cap of the individual sample container which would indicate tampering if removed. In addition, all edges of the cooler lid except the hinge side shall be sealed with evidence tape.

When samples are composited over a time period, unsealed samples can be transferred from one crew to the next crew. A list of samples will be made by the transferring crew and signed for by a member of the receiving crew. They will either transfer the samples to another crew or deliver them to laboratory personnel who will then acknowledge receipt in a similar manner.

Color slides or photographs taken of the sample location and of any visible pollution are recommended to facilitate identification and later recollection by the sampler. A photograph log should be made at the time the photo is taken so that this information can be written later on the back of the photo or in the margin of the slide. This log should include the signature of the photographer, time, date, site location, and brief description of the subject of the photograph. Photographs and written records, which may be used as evidence, should be handled in such a way that chain-of-custody can be established.

E.4 TRANSFER OF CUSTODY AND SHIPMENT

When transferring the possession of the samples, the transferee must sign and record the date and time on the chain-of-custody record. Custody transfers, if made to a sample custodian in the field, should account for each individual sample, even when samples are transferred as a group. Every person who takes custody must fill in the appropriate section of the Chain-of-Custody Record. To prevent undue proliferation of custody records, the number of custodians in the chain-of-possession should be as few as possible.

The field custodian, or field inspector if a custodian has not been assigned, is responsible for properly packaging and dispatching samples to the appropriate laboratory for analysis. This responsibility includes filling out, dating, and signing the appropriate portions of the Chain-of-Custody Record. A Chain-of-Custody Record format, containing the necessary procedural elements, is shown in Figure E-1.

All packages sent to the laboratory should be accompanied by the Chain-of-Custody Record and other pertinent forms. A copy of these forms should be retained by the originating office (either carbon or photographic copy).

Figure E-1. Sample Chain of Custody Record

ZYX Engineers

CHAIN OF CUSTODY RECORD - USAEC SAMPLES

COC By: _____

Installation (2):
Sample Program (3):
Laboratory (2):

ZYX Field Sampler Initials (3)	Time of Sampling (Military Clock) (4)	Site ID (10)	ZYX Field Sample ID (8)	File Type (3)	Site Type (4)	Depth Ft. (5)	Sample Techniques (1)	Sample Flag Code (1)	Sample QC Code (1)	TEST NAME / CODE	Matrix	Number of Containers

Relinquished by: ZYX Engineers
Date: _____ Time: _____ Signature: _____

Accepted by:
Date: _____ Time: _____ Signature: _____

Relinquished by:
Date: _____ Time: _____ Signature: _____

Accepted by:
Date: _____ Time: _____ Signature: _____

Carrier: _____ Air Bill Number: _____

Comments:

Total No. of Containers

Mailed packages can be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent chain-of-custody documentation. Any other commercial carrier transmittal documents shall also be maintained with the permanent chain-of-custody documentation.

Samples to be shipped must be so packed as not to break and the package so sealed or locked that any evidence of tampering may be readily detected. Custody seals are narrow strips of adhesive paper used to demonstrate that no tampering has occurred. They are intended for use on a sample transport container and for routine use on individual sample containers.

E.5 LABORATORY CUSTODY PROCEDURES

Chain-of-custody procedures are also necessary in the laboratory from the time of sample receipt to the time the sample is discarded. The following procedures are recommended for the laboratory:

- A specific person shall be designated custodian and an alternate designated to act as custodian in the custodian's absence. All incoming samples shall be received by the custodian, who shall indicate receipt by signing the accompanying custody forms and who shall retain the signed forms as permanent records.

- The sample custodian shall maintain a permanent log book to record, for each sample, the person delivering the sample, the person receiving the sample, the date and time received, the source of the sample, the sample identification or log number, how the sample was transmitted to the laboratory, the temperature of the cooler, and the condition received (sealed, unsealed, broken container, or other pertinent remarks). A standardized format should be established for log book entries. A sample receipt checklist (Appendix O) shall be used by the sample custodian as an aid in logging in the samples. A copy of the checklist shall be incorporated into the lot data package.

- A clean, dry, isolation room, building, and/or refrigerated space that can be securely locked from the outside shall be designated as a "Sample Storage Security Area."

- The custodian shall ensure that heat-sensitive, light-sensitive, radioactive, or other samples having unusual physical characteristics or requiring special handling, are

properly stored and maintained prior to analysis. It is recommended that samples for volatile analysis be stored separately from all other samples.

- Distribution of samples to individuals who are responsible for the laboratory performing the analysis shall be made only by the custodian.

- Laboratory personnel are responsible for the care and custody of the sample once it is received by them and shall be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses were completed.

- Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample should be retained in the custody room until permission to destroy the sample is received by the custodian.

- Samples shall be destroyed only after all analytical results have been validated to level 3 in the USAEC Data Management System and such action is approved by the USAEC Project Officer. Samples may be required to be held in storage longer to fulfill contractual requirements or as directed by the USAEC Project Officer.

E.6 QUESTIONS/PROBLEMS CONCERNING CUSTODY RECORDS

If a discrepancy between sample tag numbers and custody record listing is found, the person receiving custody should document this and properly store the samples. The samples should not be analyzed until the problem is resolved.

The responsible person receiving custody should attempt to resolve the problem by checking all available information (other markings or sample container, type of sample, etc.). He should then document the situation on the custody record and in his project log book and notify the project manager, quality control coordinator, and USAEC by the fastest available means, followed by a written corrective action or non-conformance report.

Changes may be written in the "Remarks" section of the custody record and should be initialed and dated. A copy of this record should accompany the written notification to the project manager and quality control coordinator.

E.7 EVIDENTIARY CONSIDERATIONS

Reducing chain-of-custody procedures as well as the various promulgated laboratory analytical procedures to writing will facilitate the admission of evidence under Rule 803(6) of the Federal Rules of Evidence (PL 93-575). Under this statute, written records of regularly conducted business activities may be introduced into evidence as an exception to the "Hearsay Rule" without the testimony of the person(s) who made the record. Although preferable, it is not always possible to have the individuals who collected, kept, and analyzed samples testify in court. In addition, if the opposing party does not intend to contest the integrity of the sample or testing evidence, admission under Rule 803(6) can save a great deal of trial time. For these reasons, it is important that the procedures following in the collection and analyses of evidentiary samples be standardized and described in an instruction manual which, if need be, can be offered as evidence of the "regularly conducted business activity" followed by the laboratory or office generating any given record.

If evidence is to be used in criminal actions, special conditions apply to use of the "Hearsay Rule." It is arguable that those portions of a sampling and analysis report dealing with matters other than sampling and analysis results come within this exception. In criminal actions, records and reports of matter observed by field investigators may not be admissible and the evidence may still have to be presented in the form of oral testimony by the person(s) who made the record or report, even though the materials come within the definition of business records. In a criminal proceeding, the opposing counsel may be able to obtain copies of reports prepared by witnesses, even if the witness does not refer to the records while testifying, and if obtained, the records may be used for cross-examination purposes.

Admission of records is not automatic under either of these sections. The business records section authorizes admission "unless the source of information or the method or circumstances or preparation indicate lack of trustworthiness," and the caveat under the public records exception reads "unless the source of information or other circumstances indicate lack of trustworthiness."

APPENDIX F

SARM REPOSITORY PROGRAM

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APPENDIX F

SARM REPOSITORY PROGRAM

F.1 SARM DEVELOPMENT

Due to the limited availability of reference materials for trace organic analyses from the NIST, USAEC has initiated a program for the development of standard analytical reference materials (SARMs) for use in its programs.

Candidate methods for high purity analyses are selected and evaluated on a preliminary basis, using known materials. Appropriate standards (traceable to NIST) are selected and procured. Sufficient analyses are run to document the random and systematic errors in the analyses. The most appropriate method of high purity analysis is selected for the evaluation of the analytical standards.

Raw materials are synthesized or procured and purified to greater than 98 mole percent. Purities above 98 mole percent can be conveniently and precisely determined in many cases by differential scanning calorimetry using the premelting technique. Wet analyses are used where required. Precision and accuracy data must be presented to support each high purity analysis used to guarantee a standard. Chromatographic analyses are used to estimate impurities and thus, support an analysis by difference. Chromatographic, spectrophotometric, and NMR examination are routinely used to ensure that each material of certified high purity is indeed the correct compound.

Each SARM is subjected to an aggravated storage period to estimate its stability. Materials showing a propensity for decomposition are repurified and stabilized if practical. If repurification and stabilization are not practical, an alternate standard must be selected. SARMs should emerge from aggravated storage with purities in excess of 98 mole percent. Any standards obtained from any other source than USAEC are not considered to be SARMs.

F.1.1. CRITERIA FOR TEST RESULTS

Results of the aggravated storage tests are expressed as mole percent purity before and after the two week test. Unanticipated observations concerning the condition of the standard are noted. Test conditions are fully documented. If the purity of the standard does not fall below the 98 mole percent value and there are no conditions observed in the standard that would interfere with the analytical system, the standard passes the test.

F.1.2. TEST PROCEDURE

Liquid SARMs are sealed in glass bottles with crimp-type septum tops or glass ampules, while solid SARMs are sealed in screw top bottles. The SARMs are sealed under normal atmosphere and stored at 70°C for 2 weeks. These SARMs are then cooled and stored in a freezer until they can be analyzed. If a standard degrades below 98 mole percent, the cause is sought and special storage conditions are developed. Special storage conditions might include dark glass containers, inert atmosphere, lowered temperature, or addition of a stabilizer. If a material is found to be too unstable for storage, a new SARM is selected. The analytical technique initially used to guarantee the purity of each new SARM is repeated after aggravated storage in order to detect degradation.

F.1.3. REPORTS

The results of aggravated storage tests are submitted to the USAEC Chemistry Branch. The Chemistry Branch reviews the suitability of each material and all its supporting data for adequacy as a SARM.

F.2. SARM SURVEILLANCE PROGRAM

At six-month intervals, surveillance samples are removed from the repository and reanalyzed by the original acceptance methods.

F.2.1. PURPOSE

The purpose of this surveillance program is to confirm the integrity of each SARM by scheduled analyses.

F.2.2. CONDITIONS

All SARMs are protected from UV radiation and stored in bulk at 4°C. SARMs which have been purchased at 98 mole percent purity are stored in the manufacturer's container. Where possible, purified SARMs are stored in glass stoppered flasks which have been sealed with Parafilm. Air sensitive compounds are stored under inert atmosphere. Hygroscopic compounds are stored with desiccant in a sealed outer container.

F.2.3. TEST PROCEDURE

A specimen is withdrawn (under the appropriate atmosphere) from each SARM at prescribed intervals. Purities of these specimens are determined using the original acceptance methods.

F.2.4. CRITERIA FOR SURVEILLANCE

The standards must remain at least 98 mole percent pure through the surveillance program. If a SARM fails to meet this criterion, its use is suspended immediately and all laboratories using it are notified by the central repository by phone.

F.2.5. PROGRAM

The surveillance program for each SARM begins when the material is purified and placed in the 4°C repository. If further purification is indicated by the aggravated storage phase, the surveillance period is reinitiated upon completion of the repurification. Thus, the aggravated storage is carried out concomitantly with the first 2 weeks of the first surveillance cycle. Any required subsequent repurification of the SARM reinitiates the surveillance program. Each surveillance cycle lasts 6 months. The entire program continues for 2 years for each SARM. After 2 years, aggravated storage will be repeated on a specimen of the original materials or newly obtained material as availability and projected needs for the material at that time dictate. Materials which have been deleted from the surveys will be removed from the surveillance program at the convenience of USAEC.

F.3. USER REPORTING

The user laboratory shall report any problems with received SARMs or observed degradation of any SARM immediately to the USAEC Chemistry Branch.

APPENDIX G

STANDING OPERATING PROCEDURES
LABORATORY OPERATIONS

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APPENDIX G

STANDING OPERATING PROCEDURES
LABORATORY OPERATIONS

The laboratory shall have written standing operating procedures (SOPs) for all procedures and methods. SOPs shall be available for the following areas and shall contain, at a minimum, the information described:

- Training-- These SOPs describe the training procedures used by the laboratory to ensure that personnel are qualified to perform the required analyses.

- Sample Receipt and Logging -- These SOPs describe the precautions to be used in opening sample shipment containers, as well as procedures used to verify that chain-of-custody has been maintained, to examine samples for damage, to check for proper preservatives and temperature, to assign the testing program, and to log samples into the laboratory sample streams.

- Sample and Extract Storage -- These SOPs describe the storage conditions for all samples, procedures used to verify and document daily storage temperature, and procedures used to ensure that custody of the samples is maintained while in the laboratory.

- Sample Scheduling -- These SOPs describe the procedures and criteria used for scheduling work in the laboratory, including procedures used to ensure that holding times or contract analytical/reporting requirements, if applicable, are met.

- Preventing Sample Contamination -- These SOPs describe the procedures that will be used to prevent cross contamination or lab contamination of samples and extracts.

- Security for Laboratory and Samples -- These SOPs describe the procedures for ensuring that equipment or samples in the laboratory are not tampered with and the limit of access to authorized personnel only.

- Traceability/Equivalency of Standards -- These SOPs describe the

procedures for the obtaining of standards and their inventory and the methods to be employed for the characterization of non-SARMs and the demonstration of equivalency for secondary standards.

• Standard Solution Verification -- These SOPs detail the procedures used to prepare, verify, and document every standard and reagent solution, including reagent-grade water, used in the laboratory. Information concerning specific grades of materials used in the preparation, appropriate glassware and containers for preparation and storage, labeling and recordkeeping for stocks and dilutions, procedures used to verify concentration and purity, and safety precautions to be taken should be included in the SOPs.

• Maintaining Instrument Records and Logbooks -- These SOPs describe procedures used to ensure that laboratory equipment and instrumentation are in working order. The SOPs describe calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, service contracts or service arrangements for all equipment, and spare parts available in-house. Calibration and maintenance of laboratory equipment and instrumentation shall be in accordance with manufacturers' specifications and shall be documented.

• Sample Analysis and Data Control Systems -- These SOPs describe procedures that are used for the operation of the sample analysis and data control systems.

• Glassware Cleaning -- These SOPs describe the procedures that are used in the cleaning of glassware used in the laboratory.

• Technical and Managerial Review of Laboratory Operations and Data Package Preparation -- These SOPs describe the procedures that are used to ensure that operations are being carried out according to requirements, in a timely manner and the interaction between management and the laboratory staff.

• Internal Review and Contractually Required Quality Assurance and Quality Control Data for Each Individual Data Package -- These SOPs detail the type, purpose, and frequency of QC samples analyzed in the laboratory. They should include information on the applicability of the QC sample to the analytical process, the statistical treatment of the data, and the responsibility of laboratory staff and management in generating and using the data.

• Sample Analysis, Data Handling and Reporting -- SOPs for analytical methods shall be a description of how the analysis is actually performed in the laboratory. These SOPs should include the following:

• Sample preparation and analysis procedures including applicable holding time, extraction, digestion, or preparation steps as appropriate to the method; procedures for determining the appropriate dilution to analyze; and any other information required
to perform the analysis accurately and consistently.

• Instrument standardization, including concentration and frequency of analysis of calibration standards, linear range of the method, and calibration acceptance criteria.

• Raw data recording requirements and documentation including sample identification number, analyst, data verification analyst, date of analysis and verification, and computational method(s).

• Data Reduction and Validation -- These SOPs describe the procedures used to compute analytical results from data and to review and validate the data. They should include all formulas used to calculate the results, procedures for computing and interpreting the results from QC samples, and procedures used to independently verify that the analytical results are correct. In addition, routine procedures used to monitor precision and accuracy, including evaluations of reagent, field, and trip blanks, calibration standards, control samples, duplicate and matrix spike samples, and surrogate recovery should be detailed in an SOP. The validation of data entry into the IRDMIS shall be included, i.e., check of transfer file versus input data.

• Chain-of-Custody -- These SOPs describe the procedures to be followed for controlling internal chain of custody of samples and extracts, and reporting problems of chain-of-custody from sampling contractor.

• Document Control, Including Data Package Preparation -- These SOPs describe the procedures being used to control all the data output from the analysis. They conclude the procedures for the preparation of the data package and its subsequent review.

• Corrective Action -- These SOPs describe procedures used to identify and

correct deficiencies in the analytical process. These include specific steps to take in correcting deficiencies such as preparation of new standards and reagents, recalibration and restandardization of equipment, reanalysis of samples, and additional training of laboratory personnel in methods and procedures. The SOP shall specify that each corrective action must be documented with a description of the deficiency, the corrective action taken, and the person(s) responsible for implementing the corrective action.

• Records Management -- These SOPs describe the procedures for generating, controlling, and archiving laboratory records. The SOPs should detail the responsibilities for record generation and control; policies for record retention; including type, time, security, and retrieval and disposal authorities. Records shall include:

• Project-specific records related to analyses performed for a group of samples. Project records may include an index of documents, correspondence, chain-of-custody records, request for analysis, calibration records, raw and finished analytical and QC data, data reports, and project planning documents.

• Laboratory operations records, which document the overall laboratory operation. These records may include laboratory notebooks, instrument performance and maintenance logs, software documentation, control charts, reference material certification, personnel files, laboratory SOPs, and corrective action reports.

APPENDIX H

CONTROL CHART CONSTRUCTION

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APPENDIX H

CONTROL CHART CONSTRUCTION

H.1 SINGLE DAY $\bar{x}$ - R CONTROL CHARTS

Control charts are prepared for each control analyte using data from the duplicate spiked QC samples in each lot to determine percent recovery:

$$\frac{\text{*Found Concentration}}{\text{Spiked Concentration}} \times 100$$

(* Method Blank correction addressed in Section 9.4). Use of percent recovery allows for minor variations in spiking solution concentrations.

To prepare control charts, the analyst should have access to the following data:

- Percent recovery of each analyte in the two high concentration spiked QC samples (Class 1);

- Average ($\bar{x}$) percent recovery for the two spiked QC samples (Class 1) in each lot; and

- Difference (R) between the percent recoveries for the two spiked QC samples (Class 1) in each lot.

The initial control chart shall be prepared using the first four days of analysis data closest to the spiking concentration used during analyses. The average $\bar{x}$ ($\bar{\bar{x}}$), average range (R), and control limits for $\bar{x}$ and R shall be updated after each in-control lot for the first 20 lots. Limits established after lot 20 shall be used for the next 20 lots. Control charts shall be updated after each 20 lots, thereafter, using the most recent 40 points. In interpreting the control charts developed for the initial lots (lots 1-20), the limits established from the previous lots will be used to control the current lot. When modified limits (see Appendix L) are established, data for samples will be accepted if the control data falls between the modified limits. If modified limits have not been established, data

for samples will be accepted based on the recoveries established during validation and the current performance of the method. In updating the control charts, the new data must be combined with the individual values of previous average percent recoveries and not the mean of all previous data. Only lots evaluated as in-control are applicable to the 20 and 40 lot requirements for establishing and updating control limits. Out-of-control or outlier points should be plotted; however, such lots are not utilized in lot number requirements or control limit calculations.

The formulae used to establish and maintain control charts for duplicates are as follows:

Average: $\bar{\bar{x}} = \frac{\sum \bar{x}}{K}$

Range: $\bar{R} = \frac{\sum R}{K}$

where:

$\bar{\bar{x}}$ = between group average of the pairs (within group) average recovery;

$\bar{x}$ = average within group recovery for data pairs;

R = within group difference between recoveries for data pairs; and

K = cumulative number of pairs in data base.

UWL on Average*: $UWL_{\bar{x}} = \bar{\bar{x}} + 1.25\ \bar{R}$

UCL on Average: $UCL_{\bar{x}} = \bar{\bar{x}} + 1.88\ \bar{R}$

LWL on Average*: $LWL_{\bar{x}} = \bar{\bar{x}} - 1.25\ \bar{R}$

LCL on Average: $LCL_{\bar{x}} = \bar{\bar{x}} - 1.88\ \bar{R}$

UWL on Range: $UWL_R = 2.511\ \bar{R}$

UCL on Range: $UCL_R = 3.267\ \bar{R}$

LWL on Range: $LWL_R = 0$

LCL on Range: $LCL_R = 0$

One possible format for maintaining $\bar{x}$ - R chart data in both tabulated and graphic form is shown in Figures 11-1 and 11-2. Examples of $\bar{x}$ - R data and charts are provided in Appendix L.

* See Appendix L for discussion on Modified Limits

All recoveries shall be plotted, whether or not the lot is in-control. Plotted points represent averaged instrument measurements and not the individual measurement values. Each individual recovery measurement value shall be tested as an outlier using Dixon's Test at the 98 percent confidence level (Appendix K). If the datum is not classified as an outlier by the test, the point shall be included in updating the control chart limits. If the datum is classified as an outlier, it shall not be used by the program in updating the control chart limits. Method control shall be judged according to the criteria in Section 8.7. Range data are not subject to outlier testing.

After the first 20 in-control sample lots, control limits shall be recalculated using only in-control data points. The control limits shall then be drawn backward to encompass all previous points. Any points falling outside the control limits (UCL or LCL) shall be dropped and the control limits recalculated using only points between the UCL and LCL. This practice of dropping points and recalculating limits is only performed once. Charts will then be updated with the newly calculated control limits and all points plotted. Lots associated with points outside the new control limits may require resampling and/or reanalysis as determined by the USAEC Project Officer on a case-by-case basis. These limits shall then be used to control analysis of the next 20 lots. Once 60 or more lots are analyzed by a particular method, control limits are recalculated based upon the 40 most recent in-control lots, i.e., control limits for the 60th lot are based on lots 21-60 (40-point slide).

Figure H-1. Sample $\bar{x}$ - R CONTROL CHART DATA TABULATION FORMAT

Laboratory Quality Control Worksheet -- $\bar{X}$ - R Chart

Laboratory _____ Date _____

Method of Test or Operation _____

Reference Value _____ Increment of Measurement _____

Data

Date	No.	X_1	X_2	X_3	$\bar{X}$	R

Totals $\Sigma \bar{X}$ _____ Σ R _____

X_i = observed value R = largest - smallest

K = sets of values CL = control limit

Σ = summation WL = warning limit

U = upper L = lower

D_4 = 3.267 for n = 2; 2.575 for n = 3

A_2 = 1.880 for n = 2; 1.023 for n = 3

Calculations

1. $\bar{R} = \Sigma R \div K$

   ___ = ___ ÷ ___

2. $UCL_R = D_4 \times \bar{R}$

   ___ = ___ x ___

3. $UWL_R = 2/3(D_4\bar{R} - \bar{R}) + \bar{R}$

   ___ = 2/3(___ - ___) + ___

4. $\bar{\bar{X}} = \Sigma \bar{X} \div K$

   ___ = ___ ÷ ___

5. $CL_{\bar{X}} = A_2 \times \bar{R}$

   ___ = ___ x ___

6. $WL_{\bar{X}} = 2/3 \times CL_{\bar{X}}$

   ___ = 2/3 x ___

7. $UCL_{\bar{X}} = \bar{\bar{X}} + CL_{\bar{X}}$

   ___ = ___ + ___

8. $UWL_{\bar{X}} = \bar{\bar{X}} + WL_{\bar{X}}$

   ___ = ___ + ___

9. $LWL_{\bar{X}} = \bar{\bar{X}} - WL_{\bar{X}}$

   ___ = ___ - ___

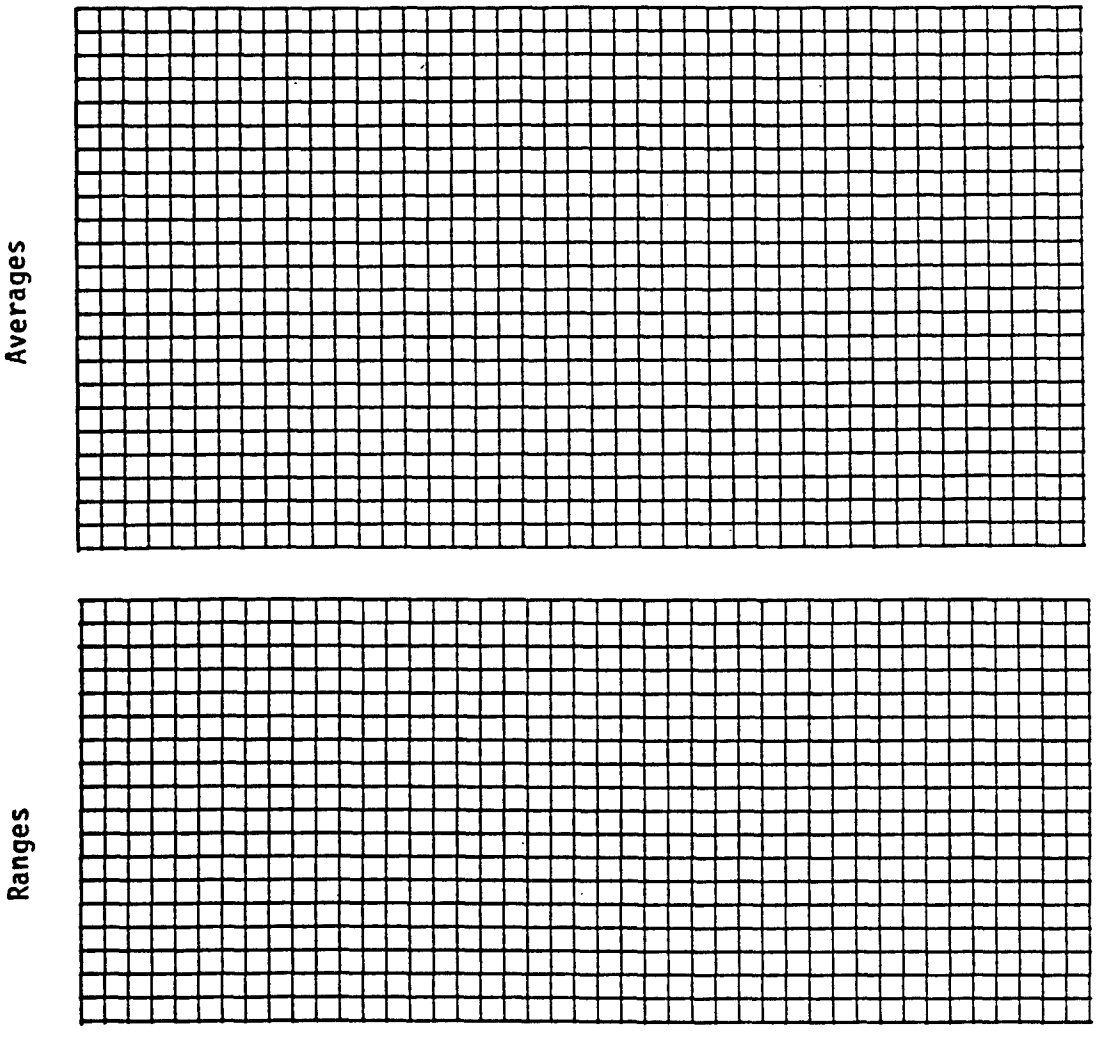
10. $LCL_{\bar{X}} = \bar{\bar{X}} - CL_{\bar{X}}$

    ___ = ___ - ___

Figure H-2. Sample $\bar{x}$ - R CONTROL CHART PLOTTING FORMAT

Laboratory Quality Control Worksheet -- $\bar{X}$ - R Chart

Operation _____ Date _____



Directions:

1. Draw $\bar{R}$ line _____
2. Draw UCL_R line _____
3. Draw UWL_R line _____
4. Plot R's as generated
5. Draw $\bar{\bar{X}}$ line _____
6. Draw $UCL_{\bar{X}}$ line _____
7. Draw $UWL_{\bar{X}}$ line _____
8. Draw $LWL_{\bar{X}}$ line _____
9. Draw $LCL_{\bar{X}}$ line _____
10. Plot $\bar{X}$'s as generated

If the method is judged to be out-of-control (Section 8.7) and reanalysis occurs, no point from the initial analysis may be used to update charts.

H.2 THREE-POINT MOVING AVERAGE CONTROL CHARTS

Moving average control charts shall be maintained for each control analyte spiked in the single low concentration spiked QC sample (Class 1) The X - R three-point moving average control chart shall be constructed for each control analyte as follows:

- Use percent recovery to allow for minor variations in spiking concentration;

- The first plotted point is the average of the first three recoveries (from certification, at concentrations nearest the spiking level);

- Subsequent points are obtained by averaging the three most recent individual recovery values (outliers excluded from calculation, but not from plot);

- The range for each point is the difference between the highest and lowest value for each group of three values; and

- The central line, UWL, UCL, LWL, and LCL for the control charts are calculated using the following formulae:

Average: $\bar{\bar{x}} = \frac{\sum \bar{x}}{K}$

Range: $\bar{R} = \frac{\sum R}{K}$

where:

$\bar{\bar{x}}$ = between group average of the three points (within group) average recovery;

$\bar{x}$ = average within group recovery for the three points;

R = within group difference between recoveries for data sets; and

$\bar{R}$ = between group average of the three points (within group) average range

K = cumulative number of sets in data base.

UWL on Average*: $UWL_{\bar{x}} = \bar{\bar{x}} + 0.682\ R$

UCL on Average: $UCL_{\bar{x}} = \bar{\bar{x}} + 1.023\ \bar{R}$

LWL on Average*: $LWL_{\bar{x}} = \bar{\bar{x}} - 0.682\ \bar{R}$

LCL on Average: $LCL_{\bar{x}} = \bar{\bar{x}} - 1.023\ \bar{R}$

UWL on Range: $UWL_R = 2.050\ \bar{R}$

UCL on Range: $UCL_R = 2.575\ \bar{R}$

LWL on Range: $LWL_R = 0$

LCL on Range: $LCL_R = 0$

All data shall be plotted, whether or not the lot is in-control. Plotted points represent averaged instrument measurements and not the individual measurement values. Each individual recovery measurement value shall be tested as an outlier using Dixon's Test at the 98 percent confidence level (Appendix I). If the datum is not classified as an outlier by the test, the point shall be used by the program to update the control chart limits. If one of the individual measurements is an outlier, it shall be used in calculating the three-point moving average for plotting only, but is then excluded from calculations which are based on the three most recent acceptable individual points and the control chart limits determined accordingly. Method control shall be judged according to the criteria in Section 8.7. Range data are not subject to outlier testing.

After the first 20 in-control sample lots, control limits shall be recalculated using only in-control data points. The control limits shall then be drawn backward to encompass all previous points. Any points falling outside the control limits (UCL or LCL) shall be dropped from the calculations (but left on the charts) and the control limits recalculated using only points between the UCL and LCL. This practice of dropping points and recalculating limits is only performed once. Charts will then be updated with the newly calculated control limits and all points plotted. Lots associated with points outside the

new control limits may require resampling and/or reanalysis as determined by the USAEC Project Officer on a case-by-case basis. These limits shall then be used to control analysis of the next 20 lots. A maximum of the 40 most recent lots will be used to recalculate control limits for 60 or more lots (40-point slide).

An example of data tabulation and plotting using moving average $\bar{x}$ - R charts is shown in Appendix K.

APPENDIX I

OUTLIER TEST

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APPENDIX I

OUTLIER TEST

An extreme observation (outlier) is a datum that appears to be different from the main data pattern. Such observations may be caused by the following:

- A measurement that was read, recorded, or transcribed incorrectly;
- A faulty instrument;
- Incorrectly prepared standards;
- Incorrect calculations;
- Incorrect application of an analytical method;
- Degradation of standard or spiking solutions;
- Environmental conditions that have changed significantly; or
- Other unidentified instrumental problems.

The principal safeguards against obtaining or using an outlier are vigilance during all operations and visual inspection of data before performing statistical analyses.

If a datum falls above or below the control limits of either the X or R control chart or if identified as an outlier by Dixon's test, the value shall be investigated. Sometimes the investigation will reveal a recording or computational mistake that can be revised to obtain the correct value. If an error is found but the correct value cannot be determined, the erroneous value shall not be used in statistical calculations. When errors are found, either correctable or uncorrectable, all analytical results for that lot must be inspected to ensure that erroneous results are not reported. If an uncorrectable error affected results of environmental samples, the lot shall be judged as out-of-control and analyses must be repeated.

DIXON'S TEST

Dixon's test expresses the gap between an outlier and the nearest value as a fraction of the range between the smallest and largest value.

The entire data set must be ordered from highest to lowest, with the highest value assigned a rank of 1 (X_1) and the lowest value a rank of n (Xn). The test criterion (r) varies with sample size, as follows:

- For less than eight measurements, reject Xn (the lowest value) if

$$\frac{Xn-X(n-1)}{Xn - X1} > r(10);$$

- For less than eight measurements, reject X1 (the highest value) if

$$\frac{X2-X1}{Xn-X1} > r(10);$$

- Between eight and ten measurements, reject Xn (the lowest value) if

$$\frac{Xn-X(n-1)}{Xn-X2} > r(11);$$

- Between eight and ten measurements, reject X1 (the highest value) if

$$\frac{X2-X1}{X(n-1)-X1} > r(11);$$

- Between eleven and thirteen measurements, reject Xn (the lowest value) if

$$\frac{Xn-X(n-2)}{Xn-X2} > r(21);$$

- Between eleven and thirteen measurements, reject x1 (the highest value) if

$$\frac{X3-X1}{X(n-1)-X1} > r(21);$$

- Over thirteen measurements, reject Xn (the lowest value) if

$$\frac{Xn-X(n-2)}{Xn-X3} > r(22);$$

- Over thirteen measurements, reject X1 (the highest value) if

$$\frac{X3-X1}{X(n-2)-X} > r(22).$$

The critical values for the test statistic at 98 percent confidence level are shown in Table I-1. If the test statistic is greater than the critical value from the Table, then the data point is an outlier. Once adequate data are available, n shall be kept constant at 20, with the 20 most recent data points being used.

Table I-1. CRITICAL VALUES FOR DIXON'S OUTLIER TEST

Number of Measurements (n)	Criterion (r)	Critical Value of r (a = 0.02)	Critical Value of r (a = 0.05)
3		0.988	0.970
4		0.889	0.829
5	r_{10}	0.780	0.710
6		0.698	0.625
7		0.637	0.568
8		0.683	0.615
9	r_{11}	0.635	0.570
10		0.597	0.534
11		0.579	0.625
12	r_{21}	0.642	0.592
13		0.615	0.565
14		0.641	0.590
15		0.616	0.568
16		0.595	0.548
17		0.577	0.531
18		0.561	0.516
19		0.547	0.503
20	r_{22}	0.535	0.491
21		0.524	0.480
22		0.514	0.470
23		0.505	0.461
24		0.497	0.452
25		0.489	0.445

APPENDIX J

$\bar{x}$ - R CHART DATA TABULATION AND GRAPHING FOR DUPLICATE SPIKE RECOVERY

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Figure J-1

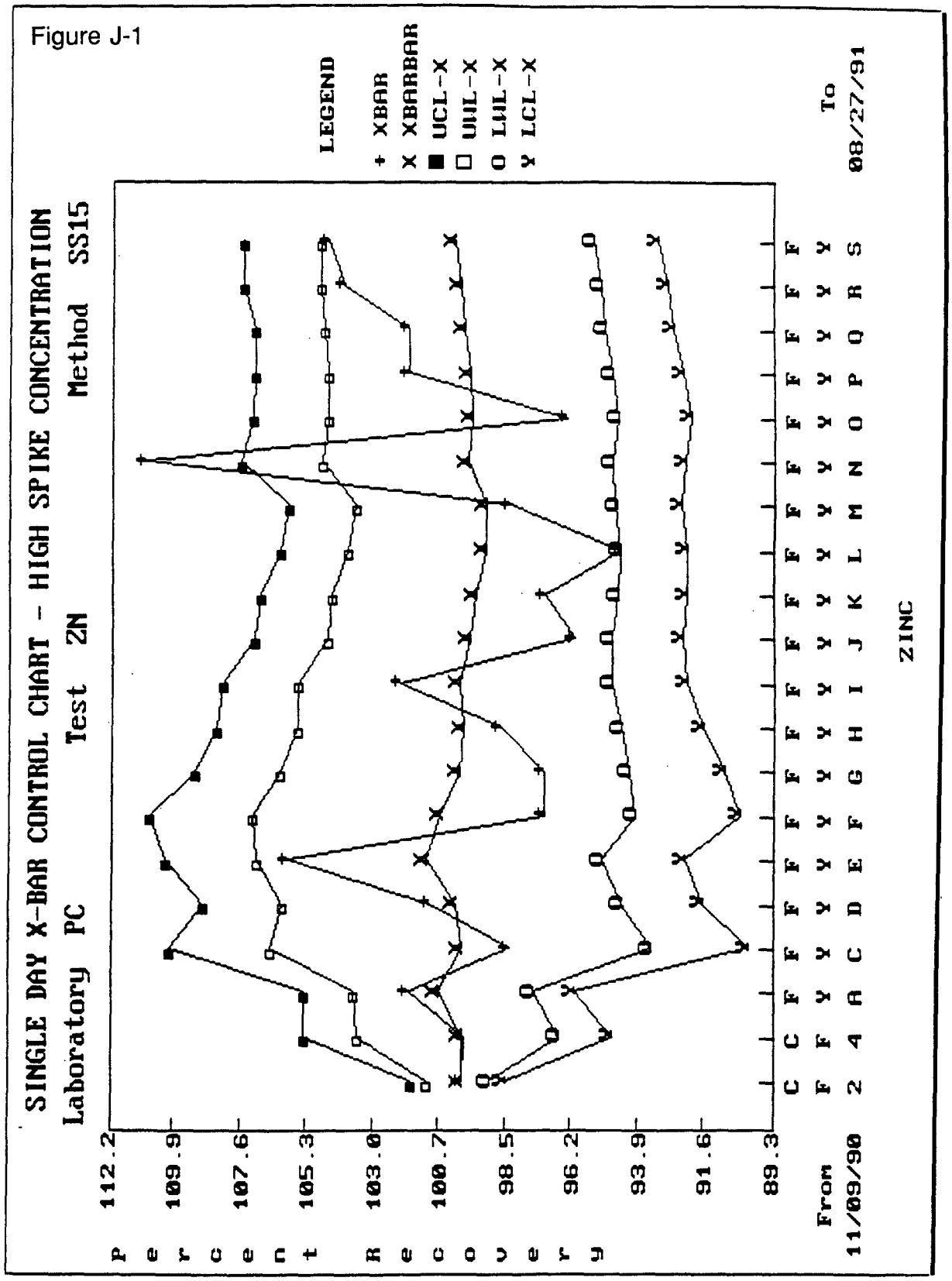
SINGLE DAY X-BAR CONTROL CHART - HIGH SPIKE CONCENTRATION
Laboratory PC Test 2N Method SS15
LEGEND
+ XBAR
X XBARBAR
■ UCL-X
□ UWL-X
O LWL-X
Y LCL-X
Percent Recovery
112.2
109.9
107.6
105.3
103.0
100.7
98.5
96.2
93.9
91.6
89.3
From 11/09/90
To 08/27/91
ZINC

Figure J-2

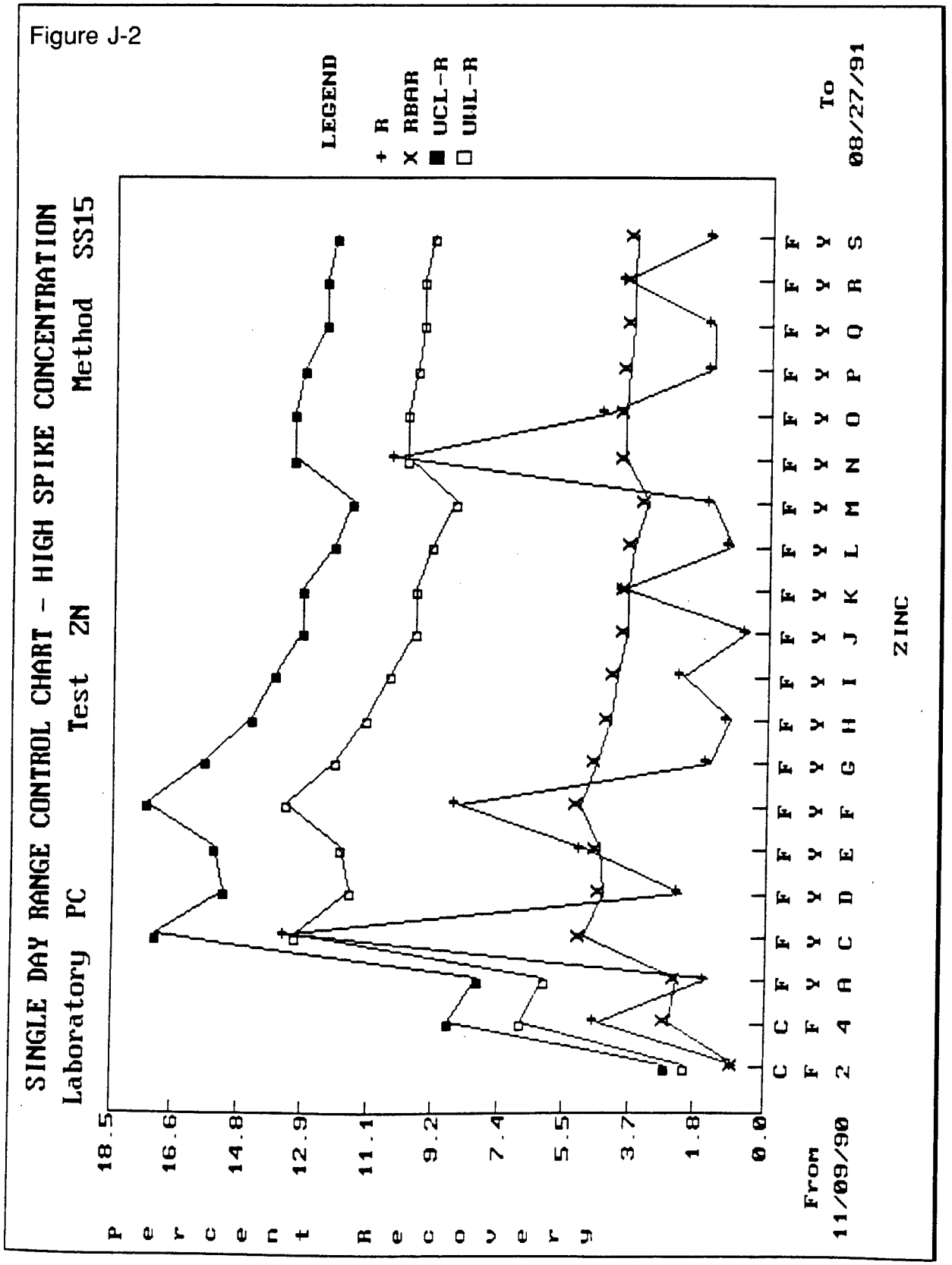
SINGLE DAY RANGE CONTROL CHART - HIGH SPIKE CONCENTRATION
Laboratory PC Test ZN Method SS15
LEGEND
+ R
X RBAR
■ UCL-R
□ UWL-R
Percent Recovery
18.5
16.6
14.8
12.9
11.1
9.2
7.4
5.5
3.7
1.8
0.0
From 11/09/90
To 08/27/91
ZINC

Figure J-3

Version 3.10

SINGLE DAY R REPORT OF PERCENT RECOVERY - HIGH CONCENTRATION
FOR ZINC

Laboratory:PC	Date:01/08/93
Method:SS15	Test Name:ZN

Date	Lot	QC Man	QC Exp	X1 Man	X1 Exp	X2 Man	X2 Exp	%X1	%X2	R	UCLR	UWLR
110990	CF2	2.00	3	2.01	3	2.00	3	100.7	99.8	0.9	2.9	2.3
110990	CF4	2.00	3	2.05	3	1.96	3	102.6	97.8	4.8	9.1	7.0
022091	FYA	1.70	3	1.75	3	1.72	3	102.9	101.2	1.8	8.2	6.3
022291	FYC	1.70	3	1.56	3	1.79	3	91.8	105.3	13.5	17.3	13.3
030691	FYD	1.70	3	1.70	3	1.74	3	100.0	102.4	2.4	15.4	11.8
031391	FYE	1.70	3	1.76	3	1.85	3	103.5	108.8	5.3	15.7	12.1
040391	FYF	1.70	3	1.58	3	1.73	3	92.9	101.8	8.8	17.6	13.6
040491	FYG	1.70	3	1.64	3	1.67	3	96.5	98.2	1.8	16.0	12.3
041191	FYH	1.70	3	1.67	3	1.69	3	98.2	99.4	1.2	14.7	11.3
041891	FYI	1.70	3	1.72	3	1.76	3	101.2	103.5	2.4	14.0	10.8
042291	FYJ	1.70	3	1.64	3	1.63	3	96.5	95.9	0.6	13.1	10.0
060391	FYK	1.70	3	1.69	3	1.62	3	99.4	95.3	4.1	13.1	10.0
060491	FYL	1.70	3	1.62	3	1.60	3	95.3	94.1	1.2	12.4	9.5
061191	FYM	1.70	3	1.66	3	1.69	3	97.7	99.4	1.8	11.8	9.0
062091	FYN	1.70	3	1.80	3	1.98	3	105.9	116.5	10.6	13.4	10.3
071991	FYO	1.70	3	1.60	3	1.68	3	94.1	98.8	4.7	13.4	10.3
072491	FYP	1.70	3	1.72	3	1.75	3	101.2	102.9	1.8	13.1	10.0
073191	FYQ	1.70	3	1.72	3	1.75	3	101.2	102.9	1.8	12.7	9.8
080691	FYR	1.70	3	1.74	3	1.81	3	102.4	106.5	4.1	12.7	9.8
082791	FYS	1.70	3	1.77	3	1.80	3	104.1	105.9	1.8	12.4	9.5

* Changes made to data

Figure J-4

Version 3.10

SINGLE DAY XBAR REPORT OF PERCENT RECOVERY - HIGH CONCENTRATION
FOR ZINC

Laboratory:PC	Date:01/08/93
Method:SS15	Test Name:ZN

Date	Lot	QC Man	QC Exp	X1 Man	X1 Exp	X2 Man	X2 Exp	%X1	%X2	XBAR	UCLX	UWLX	LWLX	LCLX	OUTLIER
110990	CF2	2.00	3	2.01	3	2.00	3	100.7	99.8	100.3	102.0	101.4	99.2	98.6	.F.
110990	CF4	2.00	3	2.05	3	1.96	3	102.6	97.8	100.2	105.6	103.8	96.8	95.0	.F.
022091	FYA	1.70	3	1.75	3	1.72	3	102.9	101.2	102.1	105.6	104.0	97.8	96.2	.F.
022291	FYC	1.70	3	1.56	3	1.79	3	91.8	105.3	98.5	110.3	106.9	93.7	90.3	.F.
030691	FYD	1.70	3	1.70	3	1.74	3	100.0	102.4	101.2	109.3	106.4	94.6	91.7	.F.
031391	FYE	1.70	3	1.76	3	1.85	3	103.5	108.8	106.2	110.4	107.4	95.4	92.4	.F.
040391	FYF	1.70	3	1.58	3	1.73	3	92.9	101.8	97.3	111.0	107.6	94.1	90.6	.F.
040491	FYG	1.70	3	1.64	3	1.67	3	96.5	98.2	97.3	109.6	106.5	94.3	91.2	.F.
041191	FYH	1.70	3	1.67	3	1.69	3	98.2	99.4	98.8	108.7	105.8	94.6	91.7	.F.
041891	FYI	1.70	3	1.72	3	1.76	3	101.2	103.5	102.4	108.5	105.8	95.0	92.3	.F.
042291	FYJ	1.70	3	1.64	3	1.63	3	96.5	95.9	96.2	107.5	105.0	95.0	92.5	.F.
060391	FYK	1.70	3	1.69	3	1.62	3	99.4	95.3	97.3	107.3	104.8	94.8	92.3	.F.
060491	FYL	1.70	3	1.62	3	1.60	3	95.3	94.1	94.7	106.5	104.2	94.7	92.3	.F.
061191	FYM	1.70	3	1.66	3	1.69	3	97.7	99.4	98.5	106.2	103.9	94.9	92.6	.F.
062091	FYN	1.70	3	1.80	3	1.98	3	105.9	116.5	111.2	107.8	105.2	95.0	92.4	.F.
071991	FYO	1.70	3	1.60	3	1.68	3	94.1	98.8	96.5	107.6	105.0	94.8	92.2	.F.
072491	FYP	1.70	3	1.72	3	1.75	3	101.2	102.9	102.1	107.5	105.0	95.0	92.5	.F.
073191	FYQ	1.70	3	1.72	3	1.75	3	101.2	102.9	102.1	107.5	105.1	95.3	92.9	.F.
080691	FYR	1.70	3	1.74	3	1.81	3	102.4	106.5	104.4	107.7	105.3	95.5	93.1	.F.
082791	FYS	1.70	3	1.77	3	1.80	3	104.1	105.9	105.0	107.7	105.3	95.8	93.5	.F.

* Changes made to data

Figure J-5

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 ---------------------------------------------
| SINGLE DAY XBAR CHARTS - HIGH CONCENTRATION |
 ---------------------------------------------

 -------------------------------------------------
| Laboratory: PC | Date: 01/08/93 | Method: SS15  |
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NOTE: This is an abbreviated report and may not reflect the entire situation. You need to examine the charts and comment on corrective measures. This program does not test for cyclical patterns.

Number of Control Analytes: 12.

Method is out-of-control.
Less than one-third of the analytes were out-of-control.
However, of this one-third, at least one analyte contained two consecutive out-of-control points.

ANALYTE	BEGIN LOT	END LOT	NUMBER OF POINTS
SE	FYR	FYS	2

The following analytes contained points classified as outliers:

ANALYTE	LOT
BE	FYI
NI	FYQ
SE	FYQ
SE	FYR

The following analytes contained points outside the UCL:

ANALYTE	LOT	XBAR	UCL
BE	FYN	101.5	100.2
BE	FYS	103.5	100.8
BA	FYN	102.4	102.1
CR	FYS	106.9	104.3
CU	FYI	103.5	100.8
CU	FYR	101.5	100.2
SB	FYS	103.5	102.5
SE	FYN	106.0	103.1
SE	FYP	104.3	104.1
SE	FYR	136.8	104.1
SE	FYS	109.0	104.5
ZN	FYN	111.2	107.8

Figure J-6

The following analytes contained points outside the LCL:

ANALYTE	LOT	XBAR	LCL
BE	FYI	49.0	89.5
BA	FYO	87.5	89.3
CO	FYL	89.2	91.8
NI	FYQ	55.4	93.4
SB	FYG	92.9	93.1
SB	FYL	91.0	92.5
SE	FYG	93.0	94.7
SE	FYJ	92.7	94.0
SE	FYL	93.3	93.6
SE	FYQ	57.9	93.5
TL	FYO	85.7	87.5

The following analytes contained seven successive points below the central line:

ANALYTE	BEGIN LOT	END LOT	NUMBER OF POINTS
SE	FYF	FYM	8

WARNING: The following analytes contained four successive points going in an upward direction:

ANALYTE	BEGIN LOT	END LOT
BE	FYP	FYS
BA	FYP	FYS
CR	FYP	FYS
PB	FYP	FYS
SB	FYP	FYS

WARNING: The following analytes contained six successive points below the central line:

ANALYTE	BEGIN LOT	END LOT
TL	FYJ	FYO

Figure J-7

SINGLE DAY RANGE CHARTS - HIGH CONCENTRATION

Laboratory: PC | Date: 01/08/93 | Method: SS15

NOTE: This is an abbreviated report and may not reflect the entire situation. You need to examine the charts and comment on corrective measures. This program does not test for cyclical patterns.

Number of Control Analytes: 12.

Method is out-of-control.
Less than one-third of the analytes were out-of-control.
However, of this one-third, at least one analyte contained two consecutive out-of-control points.

ANALYTE	BEGIN LOT	END LOT	NUMBER OF POINTS
SE	FYQ	FYR	2

The following analytes contained points outside the UCL:

ANALYTE	LOT	XBAR	UCL
BE	FYI	98.0	10.1
NI	FYQ	90.6	11.1
SE	FYQ	94.9	9.1
SE	FYR	60.3	9.1

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APPENDIX K

$\bar{x}$ - R CHART DATA TABULATION AND GRAPHING FOR THREE-POINT MOVING AVERAGE SPIKE RECOVERY

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Figure K-1

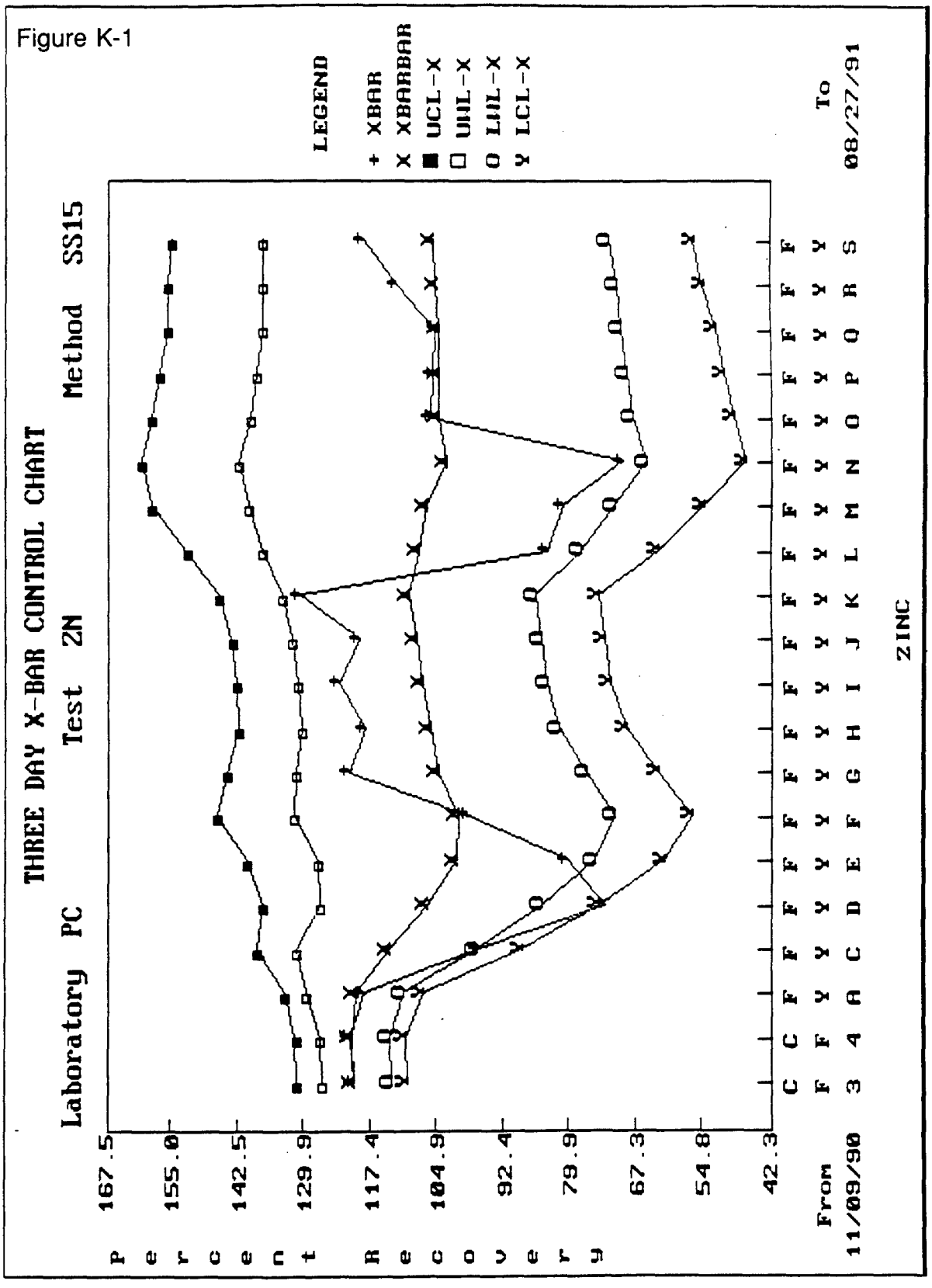
THREE DAY X-BAR CONTROL CHART
Laboratory PC Test ZN Method SS15
LEGEND
+ XBAR
X XBARBAR
■ UCL-X
□ UWL-X
0 LWL-X
Y LCL-X
Percent Recovery
167.5
155.0
142.5
129.9
117.4
104.9
92.4
79.9
67.3
54.8
42.3
From 11/09/90
To 08/27/91
ZINC

Figure K-2

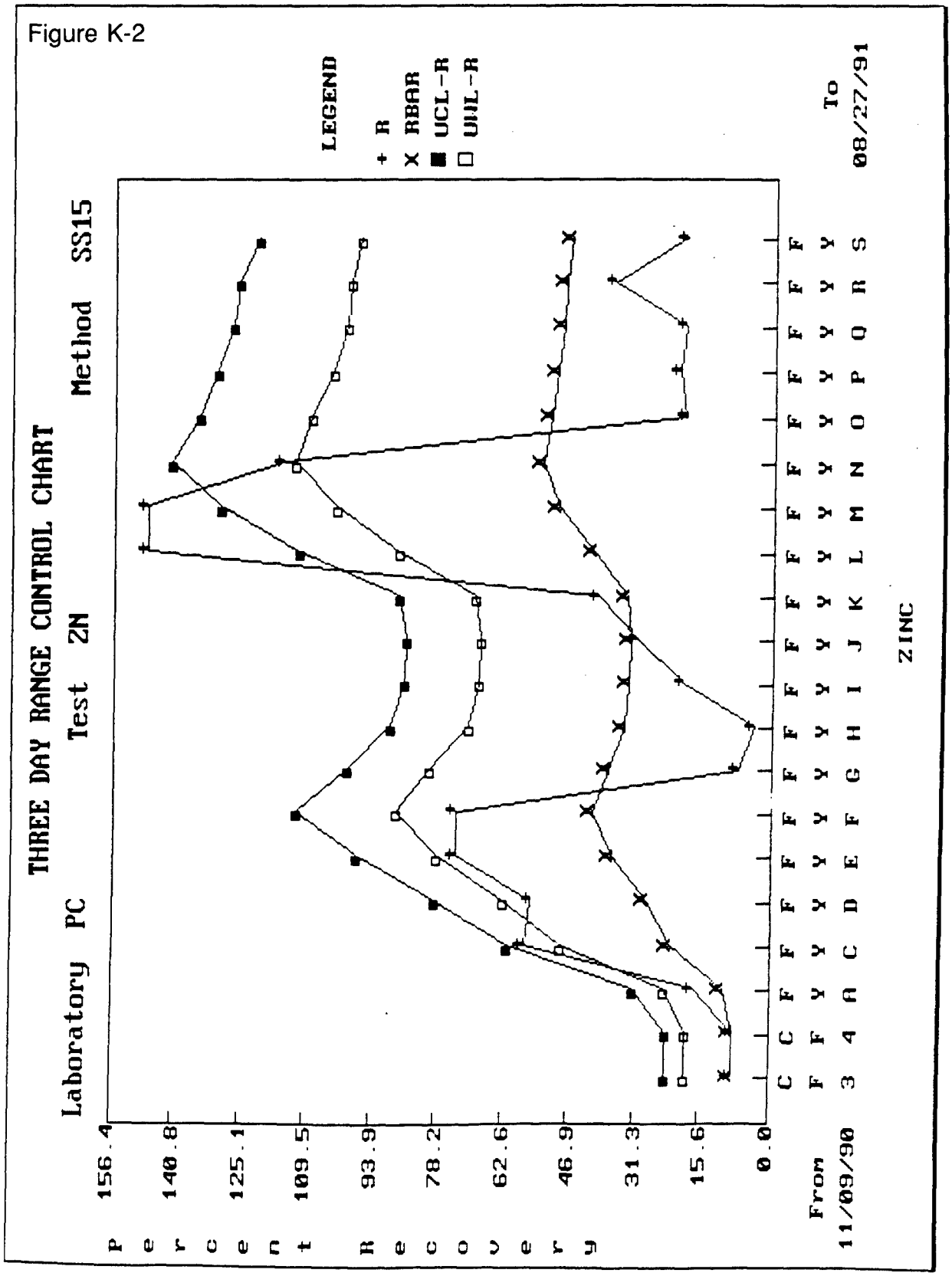
THREE DAY RANGE CONTROL CHART
Laboratory PC Test ZN Method SS15
LEGEND
+ R
X RBAR
■ UCL-R
□ UWL-R
Percent Recovery
156.4
140.8
125.1
109.5
93.9
78.2
62.6
46.9
31.3
15.6
0.0
From 11/09/90
To 08/27/91
ZINC

Figure K-3

Version 3.10

THREE DAY MOVING AVERAGE R REPORT OF PERCENT RECOVERY
FOR ZINC

Laboratory:PC	Date:01/08/93
Method:SS15	Test Name:ZN

Date	Lot	QC Man	QC Exp	X Man	X Exp	%X	R	UCLR	UWLR
110990	CF1	4.00	1	4.80	1	120.0	0.0	0.0	0.0
110990	CF2	4.00	1	4.70	1	117.5	0.0	0.0	0.0
110990	CF3	4.00	1	5.10	1	127.5	10.0	25.8	20.5
110990	CF4	4.00	1	4.90	1	122.5	10.0	25.8	20.5
022091	FYA	6.50	1	7.10	1	109.2	18.3	33.0	26.2
022291	FYC	6.50	1	4.10	1	63.1	59.4	62.8	50.0
030691	FYD	6.50	1	3.35	1	51.5	57.7	80.1	63.8
031391	FYE	6.50	1	8.30	1	127.7	76.1	99.4	79.1
040391	FYF	6.50	1	7.90	1	121.5	76.2	113.3	90.2
040491	FYG	6.50	1	7.70	1	118.5	9.2	102.0	81.2
041191	FYH	6.50	1	7.60	1	116.9	4.6	91.9	73.2
041891	FYI	6.50	1	9.00	1	138.5	21.6	88.3	70.3
042291	FYJ	6.50	1	6.90	1	106.2	32.3	87.8	69.9
060391	FYK	6.50	1	9.70	1	149.2	43.0	89.9	71.5
060491	FYL	6.50	1	1.00	-6	0.0	149.2	112.5	89.6
061191	FYM	6.50	1	6.20	1	95.4	149.2	131.8	105.0
062091	FYN	6.50	1	7.60	1	116.9	116.9	143.2	114.0
071991	FYO	6.50	1	7.00	1	107.7	21.5	137.8	109.7
072491	FYP	6.50	1	6.10	1	93.9	23.1	133.1	106.0
073191	FYQ	6.50	1	7.50	1	115.4	21.5	128.8	102.5
080691	FYR	6.50	1	8.60	1	132.3	38.5	127.2	101.3
082791	FYS	6.50	1	7.20	1	110.8	21.5	123.6	98.4

* Changes made to data

Figure K-4

Version 3.10

THREE DAY MOVING AVERAGE XBAR REPORT OF PERCENT RECOVERY
FOR ZINC

Laboratory:PC | Date:01/08/93
Method:SS15 | Test Name:ZN

Date	Lot	QC Man	QC Exp	X Man	X Exp	%X	XBAR	UCLX	UWLX	LWLX	LCLX	OUTLIER
110990	CF1	4.00	1	4.80	1	120.0	0.0	0.0	0.0	0.0	0.0	.F.
110990	CF2	4.00	1	4.70	1	117.5	0.0	0.0	0.0	0.0	0.0	.F.
110990	CF3	4.00	1	5.10	1	127.5	121.7	131.9	128.5	114.9	111.5	.F.
110990	CF4	4.00	1	4.90	1	122.5	122.5	132.3	128.9	115.3	111.9	.F.
022091	FYA	6.50	1	7.10	1	109.2	119.7	134.4	130.0	112.6	108.2	.F.
022291	FYC	6.50	1	4.10	1	63.1	98.3	140.5	132.1	98.9	90.5	.F.
030691	FYD	6.50	1	3.35	1	51.5	74.6	139.2	128.6	86.2	75.6	.F.
031391	FYE	6.50	1	8.30	1	127.7	80.8	142.4	129.2	76.6	63.4	.F.
040391	FYF	6.50	1	7.90	1	121.5	100.3	147.6	132.6	72.6	57.6	.F.
040491	FYG	6.50	1	7.70	1	118.5	122.6	145.6	132.1	78.1	64.6	.F.
041191	FYH	6.50	1	7.60	1	116.9	119.0	143.1	130.9	82.3	70.1	.F.
041891	FYI	6.50	1	9.00	1	138.5	124.6	143.5	131.8	85.0	73.3	.F.
042291	FYJ	6.50	1	6.90	1	106.2	120.5	144.4	132.8	86.2	74.6	.F.
060391	FYK	6.50	1	9.70	1	149.2	131.3	147.0	135.1	87.5	75.6	.F.
060491	FYL	6.50	1	1.00	-6	0.0	85.1	154.0	139.1	79.5	64.6	.F.
061191	FYM	6.50	1	6.20	1	95.4	81.5	159.7	142.2	72.4	54.9	.F.
062091	FYN	6.50	1	7.60	1	116.9	70.8	161.8	142.8	67.0	48.0	.F.
071991	FYO	6.50	1	7.00	1	107.7	106.7	159.7	141.5	68.5	50.3	.F.
072491	FYP	6.50	1	6.10	1	93.9	106.1	158.0	140.4	69.8	52.2	.F.
073191	FYQ	6.50	1	7.50	1	115.4	105.6	156.3	139.2	71.0	53.9	.F.
080691	FYR	6.50	1	8.60	1	132.3	113.9	156.1	139.3	71.9	55.1	.F.
082791	FYS	6.50	1	7.20	1	110.8	119.5	155.4	139.0	73.6	57.2	.F.

* Changes made to data

Figure K-5

THREE DAY XBAR CHARTS

Laboratory: PC | Date: 01/08/93 | Method: SS15

NOTE: This is an abbreviated report and may not reflect the entire situation. You need to examine the charts and comment on corrective measures. This program does not test for cyclical patterns.

Number of Control Analytes: 12.

Method is out-of-control.
Greater than one-third of the analytes were out-of-control.

LOT	NUMBER OF ANALYTES
FYL	10

The following analytes contained points classified as outliers:

ANALYTE	LOT
BA	FYJ
BA	FYK
BA	FYL
BA	FYO
CD	FYN
CO	FYL
CR	FYL
CU	FYL
NI	FYL
PB	FYL
SB	FYL
TL	FYL

The following analytes contained points outside the UCL:

ANALYTE	LOT	RECOVERY	UCL
CD	FYN	105.0	99.3
CD	FYP	105.0	99.9
CD	FYR	105.0	101.1
TL	FYQ	105.0	99.9
TL	FYS	102.0	101.0
ZN	FYK	149.2	147.0

Figure K-6

The following analytes contained points outside the LCL:

ANALYTE	LOT	RECOVERY	LCL
BE	FYA	60.0	67.3
BE	FYC	60.0	62.0
CO	FYL	0.0	66.2
CR	FYL	0.0	64.3
CU	FYL	0.0	31.1
NI	FYL	0.0	55.4
PB	FYL	0.0	72.2
SB	FYL	0.0	68.9
SE	FYL	0.0	51.8
TL	FYL	0.0	60.9
ZN	FYL	0.0	64.6

The following analytes contained two consecutive points between the UCL and UWL:

ANALYTE	BEGIN LOT	END LOT	RECOVERY	UCL	UWL	NUMBER OF POINTS
SE	FYP	FYQ	117.3	129.1	115.2	2

The following analytes contained seven successive points above the central line:

ANALYTE	BEGIN LOT	END LOT	NUMBER OF POINTS
BE	FYM	FYS	7
CD	FYG	FYS	13
NI	FYM	FYS	7

The following analytes contained seven successive points below the central line:

ANALYTE	BEGIN LOT	END LOT	NUMBER OF POINTS
SB	FYJ	FYP	7

WARNING: The following analytes contained five successive points above the central line:

ANALYTE	BEGIN LOT	END LOT
BE	FYE	FYI
CU	FYG	FYK
PB	FYE	FYI
ZN	FYE	FYI

Figure K-7

WARNING: The following analytes contained five successive points below the central line:

ANALYTE	BEGIN LOT	END LOT
CD	FYE	FYF

WARNING: The following analytes contained six successive points above the central line:

ANALYTE	BEGIN LOT	END LOT
BA	FYE	FYI
CR	FYE	FYI
CU	FYM	FYR
SE	FYN	FYS

Figure K-8

THREE DAY RANGE CHARTS

Laboratory: PC | Date: 01/08/93 | Method: SS15

NOTE: This is an abbreviated report and may not reflect the entire situation. You need to examine the charts and comment on corrective measures. This program does not test for cyclical patterns.

Number of Control Analytes: 12.

Method is out-of-control.
Greater than one-third of the analytes were out-of-control.

LOT	NUMBER OF ANALYTES
FYL	8

The following analytes contained points outside the UCL:

ANALYTE	LOT	RECOVERY	UCL
CO	FYL	90.0	57.7
CR	FYL	95.0	60.3
NI	FYL	92.7	64.6
PB	FYL	86.5	45.8
SB	FYL	101.0	93.7
SE	FYL	111.3	92.4
SE	FYM	111.3	107.4
TL	FYL	82.5	45.8
ZN	FYL	149.2	112.5
ZN	FYM	149.2	131.8

WARNING: The following analytes contained four successive points going in an upward direction:

ANALYTE	BEGIN LOT	END LOT
CU	FYE	FYE
TL	FYN	FYQ
ZN	FYI	FYL

APPENDIX L

MODIFIED LIMITS

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APPENDIX L

MODIFIED CONTROL LIMITS (MCL) FOR $\bar{x}$ CHARTS

INTRODUCTION

The ultimate goal of control charts is to help produce results of consistent and defined quality. When methods are exceptionally precise and accurate, data quality may significantly exceed requirements for the planned and use of the results (Data Quality Objectives). For example, suppose the control mean ($\bar{\bar{x}}$) is 99.5 percent with the Upper Control Limit (UCL) at 104.5 percent and the Lower Control Limit (LCL) at 94.5 percent. A lot mean of 106.0 percent would represent an out-of-control situation. However, random sampling uncertainties might suggest that recoveries between 85.0 percent and 115.0 percent would meet data quality objectives for the project. The indication of lack-of-control would not be ignored but the rejection of lot results would not be warranted.

Another important factor that applies to control charts based on duplicate spiked QC samples in each lot is that only within-day variations are reflected in the average range ($\bar{R}$) used to set upper and lower control limits on $\bar{x}$. Because lot-to-lot calibration variability is excluded from $\bar{R}$, it has been found that 10-25 percent of lot QC means will fall slightly outside of normal control limits. These minor excursions usually don't represent a true out-of-control condition and remedial action is only required when two or more successive means are outside control limits unless, of course, a mean is highly divergent.

When the average recovery differs greatly from 100 percent, many lot QC means may fail to meet data quality specifications even though reproducibility keeps these means within control limits. Alternatively, average recovery could be good but with unacceptable reproducibility. Modified Control Limits in conjunction with normal control limits on $\bar{x}$ offer a means to deal with these situations.

PROCEDURE

All previously specified steps in customary control chart establishment are followed (Appendix H). However, upper and lower warning limits are replaced by modified limits (UML $\bar{x}$ and LML $\bar{x}$) that are derived from upper and lower specification limits for individual recoveries (USL X and LSL X) using the following equations:

$$\text{UML on Average: } \text{UML } \bar{x} = \text{USL X} - M_3\bar{R}$$

$$\text{LML on Average: } \text{LML } \bar{x} = \text{LSL X} + M_3\bar{R}$$

Values for M_3 depend on the number of individual measurements in each lot mean ($\bar{x}$) and are designed to insure that each replicate measurement will be within the specification limits, except for genuine outliers. The upper and lower specification limits (USL X and LSL X) will be provided by the USAEC Chemistry Branch for those methods where a statistically valid data base has been established.

For duplicate spike QC samples (Appendix H), the equations become:

$$\text{UML } \bar{x} = \text{USL X} - 0.78\,\bar{R}$$

$$\text{LML } \bar{x} = \text{LSL X} + 0.78\,\bar{R}$$

Modified limits can also be used with moving average control charts. In contrast to duplicate spiked QC samples in each lot, $\bar{R}$ for the three-lot moving average and moving range does include lot-to-lot variability. Therefore, a high percentage of out-of-control means should not occur for measurements in a state-of-control. However, modified limits are very useful in meeting data quality specifications. For procedures with measurement capability that is superior to requirements, acceptance of lot data are facilitated for a QC moving average that is outside of control limits but within modified limits. For procedures with performance that is inadequate to meet specifications due to large $\bar{R}$ or poor accuracy, moving averages outside of modified limits command attention to improving precision and accuracy even when the averages are within current control limits.

For moving averages of n = 3 (Appendix H) the equations for modified limits are:

$$\text{UML } \bar{x} = \text{USL X} - 0.75\,\bar{R}$$

$$\text{LML } \bar{x} = \text{LML X} + 0.75\,\bar{R}$$

APPENDIX M

CONTROL CHART CHECKLIST

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APPENDIX M

CONTROL CHART CHECKLIST
(ONE WITH EACH WEEKLY SUBMISSION)

Contract/Task Number_____ Installation _____

1. The following items are included in this weekly control chart package covering method(s) _____

2. ___ Summary

3. ___ $\bar{x}$ - R Control Charts for duplicate, high or low concentration spiked QA samples, and Outlier Tests.

4. ___ $\bar{x}$ - R Three-Point Moving Average Control Charts for low concentration spiked QA samples (Class 1) and Outlier Tests.

5. ___ Observations on each chart (when applicable).

   a. ___ Trend analysis.

   b. ___ Out-of-control analysis.

   c. ___ Actions taken.

   d. ___ Demonstration of resumption of control.

6. ___ Recommendations.

7. ___ Calibration.

8. ___ Surrogate recoveries.

_____ _____
Contractor QAC Date

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APPENDIX M

INSTRUCTIONS FOR CONTROL CHART CHECKLIST

Item I. The USAEC method number(s) under which the control charts were generated that are included in this current package are to be listed in numerical order.

Item 2. A summary table shall be prepared listing the method number(s), USAEC lots, dates of analysis, and analytes that are included in this package.

Items 3 & 4. All $\bar{x}$ - R control charts generated in the control of analyses performed during this period shall be included. Each control chart shall include the following information:

- Analyte
- Method number
- Matrix
- Laboratory
- Spike concentration
- Chart title - one of the following:
  - Single Day $\bar{x}$ Control Chart
  - Single Day R Control Chart
  - Three-Point Moving Average $\bar{x}$ Control Chart
  - Three-Point Moving Average R Control Chart
- Four-letter lot designation and analysis date for each point, shown on the x-axis

- Percent Recovery (for $\bar{x}$ control charts) or Range (for R control charts) along the y-axis
- Upper control limit (UCL), on $\bar{x}$ and R control charts
- Upper warning limit (UWL), on $\bar{x}$ and R control charts
- Mean, on $\bar{x}$ and R control charts
- Lower warning limit (LWL), on $\bar{x}$ control charts
- Lower control limit (LCL), on $\bar{x}$ control charts.

The charts must contain sufficient data so that any trends, if present, could be discerned. (Charts developed during the initial stages of any analysis shall contain all points. Charts developed after the process has been stabilized, at least 20 points, shall contain at a minimum the most recent 10 points). Any point(s) that exceed the control limits shall be flagged (by circling in red) for discussion under 5b below. Any outlier tests must be included.

Item 5. The observations made during the review of the control charts, including but not limited to the items listed, shall be submitted in writing.

Item 5a. A discussion of any trends observed, the possible start of any trend, or the lack thereof, shall be included. A trend can be defined as seven points on the same side of mean, five points going in one direction or a cyclical representation of data.

Item 5b. An analysis of any points flagged on the control chart(s) as being out-of-control shall be included. Discussion should attempt to describe the cause of the out-of-control status and whether the point(s) are to be expected due to the random statistics used to demonstrate control or are the results of a possible systematic error or bias that would affect the analytical results. The discussion should include evaluation of outlier test results.

Item 5c. Describe all actions taken to get process back into control.

Item 5d. The data generated to prove that the analysis are back in control along with the criteria used ascertaining same shall be included.

Item 6. Recommendations made as to the acceptance or rejection of the lot analysis, based on Item 5. above.

Item 7. A copy of the calibration curve used for this lot.
THIS IS FOR THE FIRST LOT ONLY.

Item 8. Tables of % recovery of surrogates in all field samples, by lot and sample number. (i.e. AAAA003,004, etc.)

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APPENDIX N

CONTRACTOR QAC CHECKLIST

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APPENDIX N

CONTRACTOR QAC CHECKLIST

Before releasing data for transmission to permanent storage, for use by other project participants, or for submission via the USAEC IRDMIS, the Contractor QAC shall complete the attached checklist. One checklist shall be completed for each analytical lot. The QAC shall retain the checklist with the analytical data for the lot. The data, checklist file, and data package arranged by lot for each installation, may be inspected during any laboratory audit. The complete data/checklist file shall be forwarded to USAEC at the end of a project.

APPENDIX N

CONTRACTOR QAC CHECKLIST

Contract/Task Number _____ Installation _____

Method Number _____ Method _____

Analyte(s) _____ Lot Designation _____

No Yes		QA Program Reference	Comments
I.	Holding Times		
_ _	Extraction Time Met	6.5	
_ _	Analysis Time Met	6.5	
II.	Calibration		
A.	Initial		
1. _ _	Initial Calibration Performance	7.1.1	
4. _ _	Points Plotted	8.1.1	
B.	Daily		
1. _ _	Daily Calibration Performed	7.1.2	
2. _ _	Daily Criteria Met	7.1.2 7.5	

QA Program

No Yes		Reference	Comments
If II.B.2 is NO:			
_ _	Daily Standard Reanalyzed	7.1.2	
_ _	Daily Criteria Met	7.1.2	
_ _	Initial Calibration Performed	7.1.1	
_ _	Initial Criteria Met	8.1.1 or 8.1.3	
3. _ _	End of Day Calibration Performed	7.1.2	
4. _ _	End of Day Criteria Met	7.1.2	
If II.B.4 is NO:			
_ _	Standard Reanalyzed	7.1.2	
_ _	Criteria Met	7.1.2	
_ _	Sample Results Rejected	7.1.2	
_ _	Blow-up of manually Integrated peak(s) examined and commented on	10.5.1.2	
III. Quality Control			
A. _ _	Blank and Correct Spikes in Sample Lot	8.2	

QA Program

	No	Yes		Reference	Comments
B.	_	_	Data Plotted on Control Chart(s)	8.6	
C.	_	_	Control Points Within Limits	8.7	
If III.C is NO:					
1.	_	_	Outlier Test Performed	Appendix K	
2.	_	_	Acceptable Explanation Provided	8.7	
3.	_	_	Corrective Actions Implemented and Documented	10.0	
4.	_	_	Control Reestablished	8.7	
5.	_	_	Lot Reanalyzed	8.7	
IV.	Sample Analysis				
A.	_	_	Reported Concentrations within Certified Range	9.4	
If IV.A is NO:					
	_	_	Extracts Diluted within Range	10.4.1 or 10.4.2	
B.	_	_	All Results have Correct Significant Figures	9.4	

_____ _____

Contractor QAC Date

APPENDIX O

SAMPLE RECEIPT CHECKLIST

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APPENDIX O

SAMPLE RECEIPT CHECKLIST

	Yes	No	Comment

A. Sample Cooler

1. Is evidence tape intact?
2. Chain of Custody forms provided; filled out properly/completely?
3. Blue ice (or equiv) included __; temp recorded ___.
4. Samples intact, i.e., bottles not broken, caps in place.

B. Samples

1. Bottles labelled.
2. Labels agree with chain-of-custody form.
3. Bottles correct for type of sample.
4. Sample volume adequate for required tests.
5. Preservatives added, where required.
6. Evidence tape on bottles.

C. Log in

1. Site ID/field number entered in logbook.
2. USAEC number assigned and entered.
3. Label on bottle annotated with USAEC number.

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APPENDIX P

DATA PACKAGE CHECKLISTS

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APPENDIX P

DATA PACKAGE CHECKLISTS

Each data package will have a series of checklists associated with it as an aid in the determination of its completeness and as a means of checking compliance with USAEC requirements. These checklists will include, but are not limited to:

- Data package checklist;
- Data package document inventory list;
- Data review checklist;
- Report checklist.

The final step in the review of a data package are the signing by the QAC of the checklist and the attesting to the fact that the data are correct and defensible.

APPENDIX P

DATA PACKAGE CHECKLIST

___ Lot

_____ Method Number

I have checked this report and data package to make certain that the following conditions are in compliance with USAEC requirements:

I. GENERAL

___ 1. All enclosed copies are legible and not excessively reduced.

___ 2. There are no "yellow stickies," tablet sheets, or other undocumented forms in the data package.

___ 3. All required documents, including a completed chain-of-custody form, are enclosed.

___ 4. The data block on the outside of the data package are complete, with all other relevant information included.

II. NOTEBOOK PAGES

___ 5. All copies of notebook pages are identified by notebook number and page number.

___ 6. All units ("ug/L"; "ug/g"; "mL") are clearly defined.

___ 7. Each page has been signed and dated by the analyst and reviewer.

___ 8. All written explanations have all of the necessary information included and may stand alone as written.

III. COMPUTER DATA SHEET

___ 9. The preliminary computer data sheet has been signed and dated by both the reviewer and the analyst.

IV. CHROMATOGRAMS AND STRIP CHARTS

___ 10. All enclosed chromatograms and/or strip charts have been labelled properly, signed, and dated by the analyst.

V. CHECKLISTS

___ 11. All enclosed checklists are the current version, and have either each blank initialled or the blanks checked with a signature at the bottom of the page.

VII. CORRECTIONS

___ 12. No white-out or correction tape has been used on any raw data.

___ 13. All cross-outs consist of only a single line, and have been initialled and dated.

___ 14. All cross-outs have a legitimate, sufficient explanation alongside.

_____ _____

Analyst Signature Date Checker Signature Date

Data were obtained while the analytical process was in-control and meet the agreed upon Data Quality Objectives.

_____ _____

QAC Signature Date

APPENDIX P

DATA PACKAGE DOCUMENT INVENTORY LIST

____ _____

Lot Method Number

Analyst: If the listed document is in the data package, please initial inventory list.

_____ Review sign-off sheet;
_____ Chain-of-custody sheet, laboratory;
_____ Chain-of-custody sheet, field;
_____ Reagent blank report form;
_____ Screening chromatogram - dated and initialed by analyst;
_____ Unknown analyte report sheet;
_____ Best fit spectra for each unknown peak;
_____ NIST library search for unknowns;
_____ Coding form or approved data reporting form;
_____ Copy of extraction logbook pages;
_____ Copy of sample preparation logbook pages;
_____ Copy of analyst's notebook pages;
_____ Copy of moisture logbook pages;
_____ Copy of standards preparation (logbook pages);
_____ Raw data output - dated and initialed by analyst (printouts, etc.);
_____ DFTPP 12 hour tuning and mass calibration report(s);
_____ BFB 12 hour tuning and mass calibration report(s);
_____ Initial calibration data, including RIC, and quantitation reports for four standards;
_____ Daily calibration data, including RIC, and quantitation report;
_____ RIC and quantitation report for: field samples, QC samples, blank samples;
_____ Check standard results;
_____ Chromatogram or strip chart recorded output with analyte peak indicated, dated, and initialled by analyst;
_____ Expanded scale blow-up of manually integrated peak;
_____ Unknown report, library search, best fit spectra;
_____ Raw data for quantitated analytes (when positively identified - including difference display, and enhanced and unenhanced spectra);
_____ Example calculations.

NA - item not applicable to analytical procedure.

APPENDIX P

USAEC DATA REVIEW CHECKLIST

Lot _____ Method Number _____

HOLDING TIMES	YES	NO	N/A	COMMENTS
1. Was extraction/digestion holding time met for all samples?				
2. Was analysis holding time met for all extracts/digestates?				
3. Were all reported dilutions performed within holding times?				
PAPER TRAIL _____				
4. Is chain-of-custody information present and complete?				
5. Are all necessary forms present, complete, and filled out in blue or black ink?				
6. Are all changes made properly, and initialled/dated?				

DAILY CALIBRATION	YES	NO	N/A	COMMENTS
7. Was a standard curve for each analyte (as specified in the method) plus a blank analyzed with each daily lot?				
8. Was a new standard curve run on the day of reanalysis of diluted extracts, and was it used for sample calculation for that date?				
9. Do the calibration standards equal or bracket the concentration equivalent to the MDL and the URL (if appropriate)?				
10. Do the calibration standards equal or bracket the MDL and the highest sample or spike response in the daily lot (if appropriate)?				
11. Was the standard specified in the method reanalyzed at the end of each daily lot, and at the appropriate interval within that lot and did the response meet criteria?				
CONTROL SPIKES _____				
12. Were standard matrix control spikes (spiked with the appropriate analytes and at the designated levels) and a standard matrix blank extracted/digested and analyzed on the same date as the daily lot?				

	YES	NO	N/A	COMMENTS
13. If dilution and reanalysis have been performed on a different day, was at least one control spike reanalyzed with the diluted samples? Has this spike been reported with the data on the appropriate date?				
14. Did control spikes pass control chart criteria? If not, has an acceptable explanation been provided, and correction taken as necessary?				
SAMPLE ANALYSIS				
15. Are reported sample and control spike concentrations within the certified concentration range of the method?				
16. If sample concentrations above the URL are reported, were they diluted into the certified range with the dilution factors clearly indicated?				
17. Are reported detection limits the Method Detection Limits?				
18. Are justifications supplied for all non-use of data, analyses, etc.				
19. Are all reanalyzed samples clearly marked and explanation presented?				

	YES	NO	N/A	COMMENTS
20. Are all manual integration justified?				

QUALITY ASSURANCE REVIEWER ONLY

21. For randomly selected data points, can the reported concentrations be back calculated using the available raw data?				

REVIEWER'S SIGNATURE

CHEM: _____ DATE _____

SUPERVISOR: _____ DATE _____

QA: _____ DATE _____

APPENDIX P

USAEC REPORT CHECKLIST

Lot _____ Method Number _____

I have reviewed and checked the enclosed report for the following items:

Transcriptions

_____ 1. Soil weights and liquid volumes have been copied correctly.
_____ 2. All information from strip charts, chromatograms, and lab notebooks has been correctly transferred to the computer.
_____ 3. All information from the field chain-of-custody has been correctly copied onto the coding form.
_____ 4. Sample results and dilution factors derived from computer printouts or notebook calculations have been accurately copied onto the coding form.

Calculations

_____ 5. All calculations have been verified.
_____ 6. All reported values are uncorrected for moisture, dilution, or other factors.

Coding Form and QC Form

_____ 7. The mantissa and exponent for each sample result and dilution factor have been accurately entered onto the coding form.
_____ 8. The correct MDL has been used on the coding form.
_____ 9. The correct method ID has been noted on both the coding form and the outside of the data package.
_____ 10. Preparation date and analysis date on the coding form agree with those on the chain-of-custody.
_____ 11. The QC form indicating whether or not the QC spikes are within control has been completely and accurately completed.
_____ 12. Sample results are not reported below the MDL or above the highest standard.

_____ _____
Analyst Signature Date

_____ _____
Checker Signature Date

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APPENDIX Q

AUDIT CHECKLIST

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APPENDIX Q

LABORATORY AUDIT CHECKLIST

_____

EVALUATED LABORATORY

_____

SUBJECT PROJECT

QC Coordinator _____

Analytical Task Manager _____

Project Manager _____

Project Officer _____

Evaluator _____

Evaluation Date _____

APPENDIX Q

AUDIT CHECKLIST

	YES	NO	COMMENT
PRE-AUDIT			
1. Notified laboratory			
2. Notified project officer			
3. Made travel arrangements			
4. Reviewed background information/ data			
5. Requested laboratory to have data/ methods/personnel available			
6. Prepared agenda			
IN-BRIEFING			
7. Introduced participants			
8. Described goals and objectives of audit/agenda			
9. Identified specific areas for review that could require some laboratory preparation			
10. Discussed general overview/status on project			
11. Discussed problem areas			

	YES	NO	COMMENT
GENERAL			
12. a. Has detailed Project QC Plan (QAPjP) been submitted?			
b. Has individual been appointed as QAC who is independent from analysis?			
c. Have sufficient facilities, personnel, and instrumentation been provided to perform the required analyses?			
d. Does the QAC have the resources to function effectively?			
e. Are chemicals and reagents of sufficient quality so as not to compromise the analytical system?			
f. Is housekeeping commensurate with analytical techniques?			
g. Has a training plan been developed and training been documented?			
h. Is the correct version of USAEC supplied software being used?			

AUDIT	YES	NO	COMMENT
13. Samples chosen to follow through laboratory:			
Inorganic			
Organic			
14. Sample receiving:			
a. Are procedures/SOPs available?			
b. Are samples checked upon receipt?			
c. Is the sample checking documented?			
d. Is area secure?			
e. Are chain-of-custody forms filed?			
f. Are internal chain-of-custody forms generated?			
g. Are samples logged in according to SOP?			
h. Are USAEC numbers assigned?			
i. Are numbers allocated for QC samples?			

AUDIT (cont)	YES	NO	COMMENT
j. Are samples stored in refrigerator until needed?			
k. Is the temperature of refrigerator monitored?			
l. Is there a sign-out system for samples?			
m. Are VOA samples isolated from other samples?			
15. Inorganics Section:			
a. Are logbooks kept for:			
Digestion?			
Analysis?			
Instrument maintenance?			
Standard preparation?			
b. Are logbooks identified with unique number?			
c. Are pages of logbooks numbered?			
d. Are reagents/solvents/acids checked for purity, etc.?			

Inorganics (cont)	YES	NO	COMMENT
e. Are standards stored correctly?			
f. Is inventory of standards maintained?			
g. Are standard solutions labelled with date prepared?			
h. Are solution validity checks documented?			
i. Are standards traceable from receipt to use?			
j. Are samples maintained and stored according to SOP?			
k. Are procedures in place to minimize cross contamination?			
l. Are samples analyzed according to validated methods?			
m. Are results of analyses stored in data packages?			
16. Organics Section:			
a. Are logbooks kept for:			
Extraction?			
Analysis?			

Organics Section (cont)	YES	NO	COMMENT
Instrument Maintenance?			
Standard preparation?			
b. Are logbooks identified with unique number?			
c. Are pages in logbooks numbered?			
d. Are reagents/chemicals checked for purity, etc.?			
e. Are standards stored correctly?			
f. Is an inventory of standards maintained?			
g. Are standard solutions labelled with date prepared?			
h. Are solution validity checks documented?			
i. Are standards traceable from receipt to use?			
j. Are samples maintained and stored according to SOP?			
k. Are procedures in place to minimize cross contamination?			

Organics (cont)	YES	NO	COMMENT
l. Is tuning of GC/MS performed and documented every 12 hours?			
m. Are samples analyzed according to validated methods?			
n. Are results of analyses stored in data packages?			
17. Method selected is performed according to written validated method?			
18. Have problem areas been discussed and corrective actions reviewed/ recommended?			
19. Data Management:			
a. Data packages prepared for each lot of analysis?			
b. Data packages readily available for review?			
c. Representative data packages from each method reviewed?			
d. Data package checklists included in each package?			
Filled out correctly?			
e. Notebook pages signed and dated?			

Data Management (cont)	YES	NO	COMMENT
f. Computer print-outs readily identified?			
g. Data processing according to SOPs?			
h. Data transmittal to USAEC according to SOPs?			
20. Has data been validated according to USAEC internal SOP?			
OUTBRIEFING			
21. Summary given on findings, observations, conclusions reached?			
22. Responded to laboratory questions/ concerns?			
23. Provided forum to rectify differences between laboratory staff and audit team?			
24. Identified deficiencies and offered assistance in their correction?			
25. Copy of completed audit checklist provided to laboratory?			
26. Discussed future goals and objectives?			

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APPENDIX R

CALIBRATION/SURROGATE DOCUMENTATION

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APPENDIX R

INSTRUCTIONS FOR CALIBRATION/SURROGATE DOCUMENTATION

FOR CALIBRATION (DAILY AND CHECK STANDARD)

1. Compound - Record the compound being monitored.

2. Check the correct box - whether daily calibration standard or check standard.

3. Method No. - Record the method number of the method being used for the designated compound.

4. Concentration - Record the target or true concentration of the standard.

5. Units - Record the units of measurements.

6. Matrix - Record the matrix of the samples being determined by the assigned method number.

7. ID - Record the identity number of the standard being monitored and the USAEC lot number(s) for which the calibration is applicable.

8. Date - Record the date of the measurement.

9. Low Recovery - Record the recovery of the standard if it is lower than the low specification.

10. Low Specification Value - Record the low specification value (lowest acceptable value, i.e., either the 10 percent or 25 percent or 2 S.D. criteria) for the standard in question in the box at the top of the column. Record a recovery between the low specification value and the mean in this column.

11. Mean - Record the mean recovery (labelled recovery, if applicable) in the box at the top of the column.

12. High Specification Value - Record the high specification value (highest acceptable) value, i.e., either the 10 percent or 25 percent or 2

S.D.criteria) for the standard in question in the box at the top of the column. Record a recovery between the mean and the high spike in this column.

13. High Recovery - Record the recovery of the standard if it is greater than the high specification.

14. Comments - Record any comments on the measurement in this column.

Calibration data supporting multiple lots may be entered on the same form. A copy of the form shall be included in the data packages for the associated lots.

FOR SURROGATES:

1. Compound - Record the compound being monitored.

2. Check the box marked surr for surrogate.

3. Method Number - Record the method number of the method being used for the designated compound.

4. Concentration - Record the units of measurement.

5. Units - Record the units of measurement.

6. Matrix - Record the matrix of the samples being determined by the assigned method number.

7. ID - Record the individual sample numbers that the surrogate was spiked into.

8. Date - Record the date of the measurement.

9. Low Recovery - Record the recovery of the surrogate if it is lower than the low specification.

10. Low Specification - Record the low specification value (lowest acceptable value) for the surrogate in question in the box at the top of the column. Record a recovery between the low specification and the mean in this column.

11. Mean - Record the historical mean recovery of the surrogate in the box at the top of the column.

12. High Specification - Record the high specification value (highest acceptable value) for the surrogate in question in the box at the top of the column. Record a recovery between the mean and the high specification in this column.

13. High Recovery - Record the recovery of the surrogate if it is greater than the high specification.

14. Comments - Record any comments on the measurement in this column.

A separate form should be used of each surrogate in each lot. Only data from a single lot shall be included on a form. A copy of the form shall be included in the data package for that lot.

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APPENDIX S

FIELD SAMPLING CHECKLIST

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FIELD CHECKLIST

Signature of Auditor _____ Date of Audit _____

Project Coordinator _____ Project No. _____

Project Location _____

Type of Investigation _____
(Authority, Agency)

Briefing with Project Coordinator

Yes _ No _ N/A _ 1. Was a project plan prepared? If yes, what items are addressed in the plan?

_____
_____
_____

Yes _ No _ N/A _ 2. Were additional instructions given to project participants (i.e., changes in project plan)? If yes, describe these changes.

_____
_____
_____

Yes _ No _ N/A _ 3. Is there a written list of sampling locations and descriptions? If yes, describe where documents are.

_____
_____
_____

Yes _ No _ N/A _ 4. Is there a map of sampling locations? If yes, where is the map?

_____
_____
_____

Yes _ No _ N/A _ 5. Do the investigators follow a system of accountable documents? If yes, what documents are accountable?

_____
_____
_____

Yes _ No _ N/A _ 6. Is there a list of accountable field documents checked out to the project coordinator? If yes, who checked them out and where is this documented?

_____
_____
_____

Yes _ No _ N/A _ 7. Is the transfer of field documents (sample tags, chain-of-custody records, logbooks, etc.) from the project coordinator to the field participants documented? If yes, where is the transfer documented?

_____
_____
_____

Yes _ No _ N/A _ 8. Have the team members received the adequate training for their position? Documented?

_____
_____
_____

Yes _ No _ N/A _ 9. Have the team members received the required number of hours of OSHA training.

_____
_____
_____

FIELD CHECKLIST

FIELD OBSERVATIONS

Yes _ No _ N/A _ 1. Was permission granted to enter and inspect the facility (required if RCRA inspection)?

_____
_____
_____

Yes _ No _ N/A _ 2. Is permission to enter the facility documented? If yes, where is it documented?

_____
_____
_____

Yes _ No _ N/A _ 3. Were split samples offered to the facility. If yes, was the offer accepted or declined?

_____
_____
_____

Yes _ No _ N/A _ 4. Is the offering of split samples recorded? If yes, where is it recorded?

_____
_____
_____

Yes _ No _ N/A _ 5. If the offer to split samples was accepted, were the split samples collected? If yes, how were they identified?

_____
_____
_____

Yes _ No _ N/A _ 6. Are the number, frequency and types of field measurements, and observations taken as specified in the project plan or as directed by the project coordinator? If yes, where are they recorded?

_____
_____
_____

Yes _ No _ N/A _ 7. Are samples collected in the types of containers specified for each type of analysis? If no, what kind of sample containers were used?

_____
_____
_____

Yes _ No _ N/A _ 8. Are samples preserved as required? If no or N/A, explain.

_____
_____
_____

Yes _ No _ N/A _ 9. Are the number, frequency, and types of samples collected as specified in the project plan or as directed by the project coordinator? If no, explain why not?

_____
_____
_____

Yes _ No _ N/A _ 10. Are samples packed for preservation when required (i.e., packed in ice, etc.)? If no or N/A, explain why.

_____
_____
_____

Yes _ No _ N/A _ 11. Is sample custody maintained at all times? How?

_____
_____
_____

Yes _ No _ N/A _ 12. Is the following information completed on each chain-of-custody record?

- Sample identification number;
- Sample collector's signature;
- Date and time of collection;
- Place and address of collection;
- Waste sample description;
- Shipper's name and address;
- Name and address of organization(s) receiving sample;
- Signatures and titles of persons involved in chain-of-possession; and
- Inclusive dates of possession for each possession.

_____
_____
_____

Yes _ No _ N/A _ 13. Does a sample analysis sheet accompany all samples on delivery to the laboratory sample custodian?

_____
_____
_____

Yes _ No _ N/A _ 14. At the minimum, has the following information been completed on each sample analysis request sheet?

- Name of person receiving sample (sample custodian);
- Laboratory sample number;
- Date of sample receipt;
- Sample allocation;
- Analyses to be performed;
- Collector's name, affiliation name, address, and phone number;
- Date and time of sampling;

- Location of sampling; and
- Special handling and/or storage requirements.

_____
_____
_____

Yes _ No _ N/A _ 15. Has a field custodian been assigned for sample recovery, preservation, and storage until shipment?

_____
_____
_____

Yes _ No _ N/A _ 16. Where applicable, are sample collection containers rinsed three times with the sample material prior to collection?

_____
_____
_____

Yes _ No _ N/A _ 17. Are glass containers with Teflon-lined screw caps used to collect the following types of samples?

- Water samples for organic analyses?
- Soil and sediment samples?
- Liquid and solid hazardous waste samples (*)?

_____
_____
_____

Yes _ No _ N/A _ 18. Are polyethylene bottles with solid polyethylene-lined caps used to collect the following types of samples?

- Water samples for metal analysis?
- Water samples for pH and fluoride analysis?
- Water samples for cyanide analysis?

_____
_____
_____

Yes _ No _ N/A _ 19. Are amber glass or aluminum foil-wrapped glass bottles used for samples suspected of being photosensitive?

_____
_____
_____

* Highly alkaline wastes and wastes known to contain hydrofluoric acid should be collected in plastic containers. If it is suspected that highly alkaline materials or hydrofluoric acid is present, a small sample should be tested to determine if it reacts with the sample container.

QUALITY ASSURANCE/QUALITY CONTROL

SAMPLE DOCUMENTATION AND CHAIN-OF-CUSTODY

Yes _ No _ N/A _ 1. Is the following information being recorded in the field log book or on data sheets?

- Project name and project number;
- Purpose of sampling (e.g., quarterly sampling, resample to confirm previous analysis, initial site assessment, etc.;
- Date and time each sample was collected;
- Date and starting/stopping times (Hr:Min) for air samples;
- Date and well bailing time for groundwater;
- Blank, duplicate and split sample identification numbers;
- Sample description including type (i.e., soil, sludge, groundwater, etc.);
- Field measurement results (i.e., conductivity, pH, dissolved oxygen, combustible gas (e.g., LEL), radioactivity, etc.);
- Preservation method for each sample;
- Type and quantity of containers used for each sample;
- Weather conditions at time of sampling;
- Photographic log identifying subject, reason for photograph, date, time, direction in which photograph was taken, number of the picture on the roll;
- Sample destination;
- Analyses to be performed on each sample;
- Reference number from all forms on which the sample is listed or labels attached to the sample (i.e., chain-of-custody, bill of lading or manifest forms, etc.);
- Name(s) of sampling personnel; and

- Signature of person(s) making entries on each page.

_____
_____
_____
_____
_____
_____

Yes _ No _ N/A _ 2. Is a chain-of-custody record completed for all samples collected?

_____
_____
_____

CHECKLIST FOR MECHANICALLY CORED SAMPLES

Yes _ No _ N/A _ 1. Was the rig set up at a staked and cleared borehole location?

_____
_____
_____

Yes _ No _ N/A _ 2. Was the location, date, time, and other pertinent information recorded on boring log form?

_____
_____
_____

Yes _ No _ N/A _ 3. Was polybutyrate core tubes cut to specification and placed into core barrel?

_____
_____
_____

Yes _ No _ N/A _ 4. Was auguring and coring conducted according to the following sequence: 0-1 ft, 1-4 ft, 4-5 ft, 5-9 ft, and 9-10 ft, etc.?

_____
_____
_____

Yes _ No _ N/A _ 5. Was the core barrel removed from the borehole and opened at the completion of each coring interval?

_____
_____
_____

Yes _ No _ N/A _ 6. Was the 12-inch sections for laboratory analysis removed, capped with Teflon film lined plastic caps, sealed with tape, and immediately placed in a cooler?

Yes _ No _ N/A _ 7. Were core sections which were previously etched length-wise taped with plastic caps to prevent opening during transport to the support facility?

Yes _ No _ N/A _ 8. Were the polybutyrate line sections marked with an arrow to the top end, the boring number, and depth interval? Was a label giving the same information as well as the project name, number, the date, and the sampler's initials attached to the core in the sample handling trailer or at the site?

Yes _ No _ N/A _ 9. Were clean polybutyrate liners placed in a clean core barrel for each additional coring increment to be drilled?

Yes _ No _ N/A _ 10. Did the boring reach a predetermined depth or encounter the water table, whichever came first?

Yes _ No _ N/A _ 11. For trench disposal areas was the coring performed to the maximum depth of observable contamination?

Yes _ No _ N/A _ 12. Were all core sections transported to the support facility for logging and sample shipment preparation?

Yes _ No _ N/A _ 13. Was the boring stake left in the ground adjacent to the borehole and a board placed over the hole until it was grouted?

Yes _ No _ N/A _ 14. Were all boreholes greater than 1 ft in depth grouted the same day of construction and the borehole location stake placed in the grout?

Yes _ No _ N/A _ 15. Were one foot deep borings backfilled with native materials available adjacent to the boring?

Yes _ No _ N/A _ 16. Were the augers, and other downhole equipment decontaminated in the field prior to moving to the next borehole location upon completion of each boring?

Yes _ No _ N/A _ 17. When all borings in a specific source were completed was the drill rig initially cleaned at the source location?

Yes _ No _ N/A _ 18. Upon completion of the initial cleaning was the drill rig transported to the decontamination pad where it was thoroughly steam-cleaned before entering another source area?

Yes _ No _ N/A _ 19. Were enough augers and core barrels available so that when one set was in use a second set was being decontaminated?

Yes _ No _ N/A _ 20. At the end of the working day did all equipment, except the drill rig, and personnel proceed to the decontamination pad where decontamination procedures were initiated?

Yes _ No _ N/A _ 21. Were all bore cuttings drummed and stored while awaiting USAEC's directions for disposal?

CHECKLIST FOR HAND CORED SAMPLES

Yes _ No _ N/A _ 1. Was a piece of Teflon film and plywood placed over the top of the polybutyrate tube and the tube pushed or driven into the ground by hand?

_____
_____
_____

Yes _ No _ N/A _ 2. Was the tube removed from the ground by shovel, the tube exterior wiped clean, the ends capped with Teflon film lined plastic caps, and sealed with tape?

_____
_____
_____

Yes _ No _ N/A _ 3. Were the sample tubes marked with the boring number, the depth of the interval sampled, and the upward direction?

_____
_____
_____

Yes _ No _ N/A _ 4. Was a label containing the same information written on the sample tube as well as the project name, number, the date, and sampler's initials taped to the outside of the core?

_____
_____
_____

Yes _ No _ N/A _ 5. Were cores logged and stored in a cooler with commercially available Blue Ice prior to and during transport to the support facility sampling area where they were logged for shipment?

_____
_____
_____

FIELD CHECKLIST

DOCUMENT CONTROL

Yes _ No _ N/A _ 1. Have all unused and voided accountable documents been returned to the coordinator by the team members?

_____
_____
_____

Yes _ No _ N/A _ 2. Were any accountable documents lost or destroyed? If yes, have document numbers of all lost or destroyed accountable documents been recorded and where are they recorded?

_____
_____
_____

Yes _ No _ N/A _ 3. Are all samples identified with sample tags? If no, how are samples identified?

_____
_____
_____

Yes _ No _ N/A _ 4. Are all sample tags completed (e.g., station number, location, date, time, analyses, signatures of samplers, type, preservatives, etc.)? If yes, describe types of information recorded.

_____
_____
_____

Yes _ No _ N/A _ 5. Are all samples collected listed on a chain-of-custody record? If yes, describe the type of chain-of-custody record used and what information is recorded.

_____
_____
_____

Yes _ No _ N/A _ 6. If used, are the sample tag numbers recorded on the chain-of-custody documents?

_____
_____
_____

Yes _ No _ N/A _ 7. Does information on sample tags and chain-of-custody records match?

_____
_____
_____

Yes _ No _ N/A _ 8. Does the chain-of-custody record indicate the method of sample shipment?

_____
_____
_____

Yes _ No _ N/A _ 9. Is the chain-of-custody record included with the samples in the shipping container?

_____
_____
_____

Yes _ No _ N/A _ 10. If used, do the sample traffic reports agree with the sample tags?

_____
_____
_____

Yes _ No _ N/A _ 11. If required, has a receipt for samples been provided to the facility (required by RCRA)? Describe where offer or a receipt is documented.

_____
_____
_____

Yes _ No _ N/A _ 12. If used, are blank samples identified?

_____
_____
_____

Yes _ No _ N/A _ 13. If collected, are duplicate samples identified on sample tags and chain-of-custody records?

_____
_____
_____

Yes _ No _ N/A _ 14. If used, are spiked samples identified?

_____
_____
_____

Yes _ No _ N/A _ 15. Are logbooks signed by the individual who checked out the logbook from the project coordinator?

_____
_____
_____

Yes _ No _ N/A _ 16. Are logbooks dated upon receipt from the project coordinator?

_____
_____
_____

Yes _ No _ N/A _ 17. Are logbooks project-specific (by logbook or by page)?

Yes _ No _ N/A _ 18. Are logbook entries dated and identified by author?

Yes _ No _ N/A _ 19. Is the facility's approval or disapproval to take photographs noted in a logbook?

Yes _ No _ N/A _ 20. Are photographs documented in logbooks (e.g., time, date, description of subject, photographer, etc.)?

Yes _ No _ N/A _ 21. If film from a self-developing camera is used, are photos matched with logbook documentation?

Yes _ No _ N/A _ 22. Are sample tag numbers recorded? If yes, describe where they are recorded.

FIELD CHECKLIST

DEBRIEFING WITH PROJECT COORDINATOR

Yes _ No _ N/A _ 1. Was a debriefing held with project coordinator and/or other participants?

_____
_____
_____

Yes _ No _ N/A _ 2. Were any recommendations made to the project participants during the debriefing? If yes, list recommendations.

_____
_____
_____

Yes _ No _ N/A _ 3. Was a copy of the field checklist left with the project coordinator at the conclusion of the debriefing?

_____
_____
_____

INDEX

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USER EVALUATION SHEET/CHANGE OF ADDRESS

USAEC undertakes a continuing effort to improve the quality of its Quality Assurance Program. Your comments will aid us in achieving our goals. (Additional sheets may be attached.)

1. Organization (The following comments are provided concerning the organization of the Program).

_____

2. Useability (The following comments are provided as to the ability to find items in the Program).

_____

3. Concepts (The following comments are provided as to the existing concepts of the Program or to recommend new or innovative concepts).

_____

4. General (The following specific comments are offered for consideration in updates to the Program).

_____

5. _____
Name

_____
Organization

Current Address _____
Address

_____
City, State, Zip

_____
Telephone

6. If indicating a change of address or address correction, please provide the new or correct address in block 5 above and the old or incorrect address below.

_____
Name

_____
Organization

Old Address _____
Address

_____
City, State, Zip

_____
Telephone

Mail completed form to:

Commander
U.S. Army Environmental Center
ATTN: ENAEC-TS-C
Aberdeen Proving Ground, MD 21010-5401