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**WORK PLAN FOR  
FEASIBILITY STUDY  
MIRACLE MILE WQARF SITE**

**Prepared for  
ARIZONA DEPARTMENT OF  
ENVIRONMENTAL QUALITY**

**URS Job No. 24097087.08000  
April 2013**

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**ACRONYMS**

ADEQ	Arizona Department of Environmental Quality
ADWR	Arizona Department of Water Resources
AWQS	Aqueous Water Quality Standard
bgs	below ground surface
CAP	Central Arizona Project
COC	Contaminant of concern
Cr III	Trivalent Chromium
Cr IV	Hexavalent Chromium
FS	Feasibility Study
FWID	Flowing Wells Irrigation District
GPL	Groundwater Protection Limit
gpm	Gallons per Minute
GPS	Global Positioning Satellite
HASP	Health and Safety Plan
IDW	Investigation Derived Waste
MCL	Maximum Contaminant Level
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
NTU	Nephelometric Turbidity Units
PID	Photoionization detector
ppm	parts per million
QAPP	Quality Assurance Project Plan
RI	Remedial Investigation
RO	Remedial Objectives
SAP	Sampling and Analysis Plan
SOPs	Standard Operating Procedures
SPLP	Synthetic Precipitation Leaching Procedure
SRL	Soil Remediation Level
TCE	Trichloroethylene
URS	URS Corporation
USTS	Underground Storage Tanks
VCMHP	Villa Capri Mobile Home Park
VOC	Volatile Organic Compound
WQARF	Water Quality Assurance Revolving Fund

## **1.0 INTRODUCTION**

This Work Plan describes the site background and contaminants of concern (COCs), data gaps assessment, scope of work, community involvement, and schedule for conducting a Feasibility Study for the Miracle Mile Water Quality Assurance Revolving Fund (WQARF) Site in Tucson, Arizona. The primary COC at the site is trichloroethylene (TCE). The contents of this document are based on current knowledge of the site and anticipated field conditions; however, slight changes may be necessary if current conditions change. These conditions and changes will be discussed with and approved by the Arizona Department of Environmental Quality (ADEQ) before implementation.

### **1.1 FEASIBILITY STUDY SUMMARY**

The Feasibility Study is a process to identify a reference remedy and alternative remedies that appear to be capable of achieving remedial objectives. The potential remedies will be evaluated based on the comparison criteria to select a remedy that complies with WQARF regulations. At a minimum, two alternative remedies shall be developed for comparison with a reference remedy. At least one of the alternative remedies will employ a remedial strategy or combination of strategies that is more aggressive than the reference remedy, and at least one of the alternative remedies will employ a remedial strategy or combination of strategies that is less aggressive than the reference remedy.

The description for each remedy developed will include:

- A demonstration that the alternative will achieve the remedial objectives.
- An evaluation of consistency with the water management plans of affected water providers and the general land use plans of local governments.
- An evaluation of the comparison criteria, including:
  - The practicability of the alternative, including its feasibility, short and long-term effectiveness, and reliability,
  - The risk, including the overall protectiveness of public health and aquatic and terrestrial biota under reasonably foreseeable use scenarios and end uses of water,

- The cost of the remedial alternative, consisting of the expenses and losses including capital, operating, maintenance, and life cycle costs,
- The benefit, or value, of the remediation, and
- A discussion of the comparison criteria, as evaluated in relation to each other.

Based upon the results of the evaluation, a proposed remedy for source control and management of plume migration will be described in the Feasibility Study. The report will detail how the comparison criteria were considered and how the proposed remedy meets the requirements of Arizona Revised Statutes § 49-282.06.

## 1.2 REMEDIAL OBJECTIVES

In June 2012 ADEQ published the *Final Remedial Objectives Report*. The remedial objectives (ROs) for the site were developed as required by R18-16-406 of the remedy selection rules of the Arizona Administrative Code. These rules require that ROs be established for the current and reasonably foreseeable uses of land and waters of the state that have been or are threatened to be affected by release of a hazardous substance above a regulatory or risk-based standard.

The ROs were stated in the following terms: (1) protecting against the loss or impairment of each use; (2) restoring, replacing, or otherwise providing for each use; (3) when action is needed to protect or provide for the use; and (4) how long action is needed to protect or provide for the use.

With regards to land use, the ADEQ report states the following:

*The RO for land use at the former Spring Joint Specialists and RSC properties is to protect against possible exposure to hazardous substances in surface and subsurface soils that could occur if property improvements were made to facilitate commercial use. ADEQ will ask the property owners to place a DEUR on their properties (or portions of properties) containing hexavalent chromium above the residential SRL to ensure that current and future property owners maintain the property as non-residential use and maintain the asphalt as an engineering control. If additional work at the Site is necessary beyond maintenance of the*

*asphalt cover, ADEQ will coordinate with the property owners and work towards a remedy that is compatible with these development plans.*

With regards to water use, the ADEQ report states the following:

*The Site includes the Flowing Wells Irrigation District (FWID) which is the primary municipal water provider for the area. FWID currently operates eight wells, withdrawing approximately 2,800 acre-feet of groundwater per year for its approximately 15,000 customers. Three of these eight wells are located in or near the Site. FWID also has an inactive well in the Site, FWID-66, in which TCE was detected above the MCL and AWQS of 5 µg/L. FWID has partnered with ADEQ to construct a combination VOC and arsenic treatment system. [NOTE: The arsenic is naturally occurring and thus is not a Site COC.] This system treats the water extracted from FWID wells 70 and 75 within the Site.*

*FWID does not expect to change the number of wells or the amounts of water removed from the aquifer in the near future. However, FWID does plan to use Central Arizona Project (CAP) water to augment the groundwater supply when CAP water becomes available. This alternative is not expected to occur for several years.*

*Villa Capri Mobile Home Park (VCMHP) receives its potable water from an onsite well, though it is also connected to a municipal source for emergencies. VCMHP has 258 spaces and it is operating almost at capacity.*

The following RO was established in the ADEQ report with regards to groundwater use:

*The RO for groundwater will be to restore, replace or otherwise provide and protect for the current and future potable use of the regional aquifer threatened or impacted by TCE and/or chromium contamination emanating from the Site. This action is needed for as long as the level of contamination in the groundwater resource threatens or prohibits its use as a municipal water supply.*

Further with regards to groundwater use:

***The RO for groundwater will be to protect for the future non-potable use of the regional aquifer threatened by the TCE and/or chromium contamination emanating from the Site. This action is needed for as long as the level of contamination in the groundwater resource threatens its use as a non-potable water supply.***

*One private domestic well is located within the Site which could be used in the future. The well owner did indicate that the well is currently not used; however, in the future he would like to use the well water to irrigate his property if the groundwater becomes usable. No private domestic wells are currently being used. All other private well owners indicated that they are not using well water and do not intend to do so in the future. Therefore, no RO for domestic water supply use is needed for this Site at this time.*

Additional discussion of non-potable water use is as follows:

*Pima County Wastewater Management owns a non-exempt water supply well within the Potential Impact Area and west of the Site contaminant plume. The owner has indicated that this well is used for standby/emergency industrial non-potable water supply.*

## 2.0 DATA GAPS ASSESSMENT

As a component of development for this work plan, URS has performed a review of existing data to identify potential gaps in the hydrogeologic/contaminant characterization of the site. This included primarily a review of the Final Remedial Investigation (RI) Report (URS, 2013), which incorporated historical documents prepared by consultants for ADEQ. The data gaps evaluation also incorporated data that have been collected by URS as part of monitoring activities performed on behalf of ADEQ and the long-term groundwater monitoring program which URS has been contracted to perform. The Final Feasibility Study Report will include a description and the results of the work performed to evaluate and close these data gaps:

1. Evaluate whether perched groundwater monitor wells should be installed at the previous passive soil-gas investigation hot spots at the Friedman Recycling and Public Storage properties. New monitoring well IRA-40 will be drilled (See Appendix A – Figure 1).
2. Evaluate whether a regional aquifer monitor well is needed between the IRA-36/IRA-37 well pair and the Villa Capri Mobile Home Park well. New monitoring well IRA-38 will be drilled (See Appendix A – Figure 1).
3. Evaluate whether a regional aquifer monitor well is needed between IRA-25, IRA-24, and IRA-5. New monitoring well IRA-39 will be drilled (See Appendix A – Figure 1).
4. Evaluate whether a vertical delineation well is needed near IRA-14. New monitoring well IRA-41 will be drilled (See Appendix A – Figure 1).
5. Determine the extent of chromium contamination in the soil in the vicinity of the Spring Joint property.
6. Determine the possibility of chromium in the perched groundwater in the vicinity of the Spring Joint property.
7. Additional soil borings, soil sampling and possible installation of perched groundwater monitor wells are recommended in the areas to the north, south and east of the perched groundwater monitor wells SJ-MW-1 and SJ-MW-2 to better define the extent of the chromium contamination in this area.

(See Appendix B for #5, #6 and #7).

8. Perform additional sampling to determine whether nitrate/nitrite is a groundwater COC for the Site. If it is a COC, evaluate the extent of nitrate above the Aquifer Water Quality Standard and the source(s).

### **3.0 GROUNDWATER MODEL FINALIZATION**

The purpose of the groundwater flow model is to provide hydrologic evaluation of the various groundwater remedial strategies. The development and finalization of a three-dimensional groundwater flow model for the site will continue using MODFLOW public domain code. The model is being constructed using available data and, in areas with little data, professional judgment and published information is being used. The current version of Groundwater Vistas, a groundwater modeling environment that couples groundwater design systems with a set of comprehensive graphical analysis tools, is being used to develop, calibrate and utilize the model.

The model will be documented in a report to ADEQ. The report will detail the objectives and limitations of the model, the construction details, and the calibration results. Details will be presented graphically in a form understandable to the general public.

The fully developed model will be calibrated to historic water level information available from past monitoring at Miracle Mile WQARF site, Tucson Water, and City of Tucson Environmental Services. Following development and calibration of the groundwater flow component, particle tracking and contaminant transport modeling will be completed.

The groundwater model will be developed to simulate the regional groundwater flow conditions and the fate and transport of the TCE plume, which is the chemical of concern in the regional groundwater.

#### **3.1 MODEL LAYERING**

The groundwater model is a sub-model from the regional groundwater flow model of the Tucson active management area completed by the Arizona Department of Water Resources in 2006 which includes three layers (ADWR, 2006).

- Layer 1 simulates the Upper Tinaja beds with bottom elevation of 1950 feet (ft) mean sea level in the area of interest northeast of the highway. The bottom elevations of other areas of

Layer 1 are the same as the regional model Layer 2, which is for Tinaja Beds. The hydraulic property of Layer 1 is the same as regional model Layer 2.

- Layer 2 simulates the Middle Tinaja beds. The bottom elevations of Layer 2 are equal to the bottom elevations of regional model Layer 2 minus 300 ft. This is to include complete well screen of the FWID wells.
- The regional model Layer 3 is simulated as a confined aquifer given transmissivity without thickness. For the purpose to simulate particle tracking and fate and transport, the layer needs thickness and bottom elevations. In the sub-model, this layer was modified to have an assumed thickness of 1,000 ft. Subtracting 1,000 ft from the bottom elevations of Layer 2 generated the bottom elevations of Layer 3. The hydraulic conductivities are obtained by dividing the original transmissivity by 1,000 ft, thus are equivalent to the hydraulic property that is simulated in the regional model.

### **3.2 GROUNDWATER FLOW MODEL CALIBRATION**

Even though the groundwater conditions at the site are highly transient, a quasi-steady state flow model which represents the long-term average groundwater flow directions and groundwater flow velocity, is considered adequate for the purpose of evaluating the contaminant plume fate and transport. Thus, the groundwater flow model will be calibrated as a quasi-steady state model using the groundwater levels and pumping rates measured in 2011.

### **3.3 FATE AND TRANSPORT MODELING**

The groundwater fate and transport modeling will be conducted in two phases:

- Historical matching of the TCE plume to approximately match the current observed TCE plume extent – This process will allow estimation of the TCE source conditions and fate and transport parameters.
- Prediction of future TCE plume migration under different scenarios (such as natural attenuation and/or active remediation) using the estimated parameters and source assumptions obtained from the historical matching.

### **3.4 SOURCE ASSUMPTION**

The TCE concentrations observed in the perched aquifer have been relatively stable from 2000 to 2011. In the regional groundwater model, the contribution from the perched zone will be simulated as contaminated recharge. The TCE concentration in the recharge will be calibrated through the historical matching of the TCE plume migration.

For model predictions, the calibrated TCE recharge will be assumed as a constant source of contaminant recharge to the regional groundwater system in the near future (30 years).

### **3.5 FATE AND TRANSPORT PARAMETERS**

The fate and transport parameters include:

- Effective porosity (will be estimated based on lithology of the regional aquifer)
- Dispersivity (longitudinal, transverse, and vertical and will be estimated via historical matching of TCE plume migration)
- Soil-water partitioning coefficient
  - Total organic carbon content of saturated soil in regional aquifer
  - Bulk density of saturated soil in regional aquifer
- Biodegradation (will be estimated through data evaluation and historical matching simulation)

## **4.0 FEASIBILITY STUDY**

Based on the updated Conceptual Site Model as developed from the RI Report and recent data, a Feasibility Study (FS) will be performed with the purpose of developing a reference remedy and two alternative remedies for: a) the elevated chromium in soils; b) the TCE contaminant plume. Each remedy will consist of a combination of a remedial strategy or strategies and remedial measures that will achieve the ROs for the Site. The FS will be conducted in accordance with the Arizona Administrative Code (A.A.C.) R18 16 407.

### **4.1 WORK PLANS AND QAPPS**

Preparation and review of work plans and quality assurance project plans (QAPPs) for soil will be included. There are two work plans which can be found as the appendices to this document. Appendix A is the 2013 Monitor Well Installation Work Plan which outlines the installation and development of four new monitoring wells. There is a soil QAPP included with the Former Spring Joint Specialists Chromium Contaminated Soil Characterization Work Plan found in Appendix B. The soil QAPP outlines the procedures for sampling and analyzing the soil borings.

### **4.2 REMEDY DEVELOPMENT**

In developing the reference remedy and two alternative remedies, the remedial strategies itemized in AAC R18-16-407(F) and remedial measures presented in AAC R18-16-407(F) will be considered.

### **4.3 REMEDY COMPARISON**

The practicability, protectiveness, and cost considerations of each remedy will be evaluated as required by AAC R18-16-407(H). Where appropriate the groundwater model will be used to support such evaluation. Detailed cost estimates will be developed for each remedy to evaluate the cost considerations. Supporting documentation for the evaluation will be included in the FS Report. Criteria will be developed for the comparison of the remedies.

#### **4.4 PROPOSED REMEDY**

Based on the comparison of the reference and alternative remedies, a remedy will be proposed and the reasons for the selection will be documented. URS will submit the list of proposed remedies (remedial strategies and measures) to ADEQ for review, discussion, and approval before evaluating the proposed remedies in the FS.

#### **4.5 FEASIBILITY STUDY REPORT**

An FS Report documenting the development and comparison of a minimum of three remedies will be prepared. The FS Report will present the proposed remedy and the reasons for selecting the remedy. Associated activities such as groundwater modeling will be presented as appendices to the report.

## **5.0 COMMUNITY INVOLVEMENT**

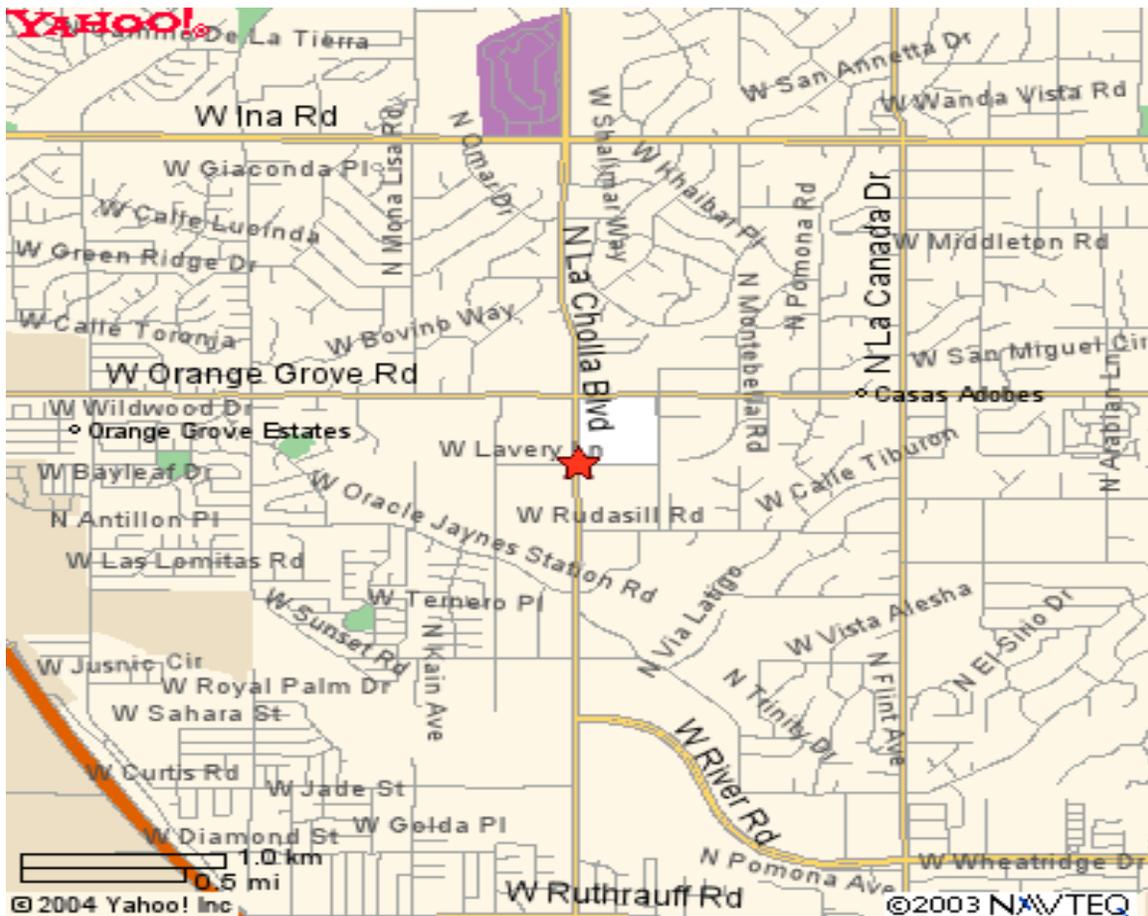
Community involvement activities for the FS will include a short presentation at an ADEQ-led Community Advisory Board meeting to summarize the results of groundwater modeling and the draft FS. Regular community correspondence provided by ADEQ will include any applicable information on the FS activities deemed appropriate by ADEQ. URS will assist ADEQ with community involvement activities, on an as-needed basis.

## **6.0 HEALTH AND SAFETY OVERVIEW**

A written Health and Safety Plan is required for hazardous waste investigations according to the Occupational Safety and Health Administration, Code of Federal Regulations 1910.120(b). In accordance with the URS health and safety program, a project-specific Health and Safety Plan (HASP) has been prepared to govern the fieldwork activities. The following table presents emergency contact information to be used for all onsite field work.

<b>Emergency Phone Numbers -</b>		
<b>Organization</b>	<b>Name</b>	<b>Phone numbers</b>
Police	Tucson Police	<b>911</b>
Ambulance	Tucson Fire Department	<b>911</b>
Hospital	Northwest Medical Center	<b>911</b>
Fire/HAZMAT	Tucson Fire Department	<b>911</b>
Poison Control Center	--	<b>(800) 332-3073</b>
URS Occupational Health Manager	Jeanette Schrimsher, RN	<b>(866) 326-7321 (24/7)</b>
URS HSE Representative	Armando Jimenez	<b>(520) 906-9177</b>
URS Regional HSE Manager	Tim Joseph	<b>(303) 740-27-67</b>

To reach the hospital from the intersection of Wetmore Road and Flowing Wells Roads, proceed north on Flowing Wells Road for three miles (Flowing Wells Road becomes La Canada Drive after crossing the Rillito Wash. At River Road, turn left (west), and go about one mile to La Cholla Blvd. Head north on La Cholla Blvd., Northwest Medical Center will be on the right just before reaching Orange Grove Road; it is at the southeast corner of Orange Grove Road and La Cholla Blvd. at 6200 N. La Cholla Blvd., Tucson, Arizona. The location of the hospital is illustrated on the following figure.



## **7.0 REFERENCES**

Arizona Department of Environmental Quality (ADEQ). 2012. *Final Remedial Objectives Report*, June.

ADEQ. 1996. *A Screening Method to Determine Soil Concentrations Protective of Groundwater Quality*, September.

Arizona Department of Water Resources (ADWR). Mason, Dale A. and Bota, Liciniu, Hydrology Division. 2006. *Regional Groundwater Flow Model of the Tucson Active Management Area Tucson, Arizona: Simulation and Application*. Modeling Report No. 13.

URS. 2013. Final Remedial Investigation Report, Miracle Mile WQARF Site, March.

**APPENDIX A**  
**MIRACLE MILE WQARF SITE**  
**2013 MONITOR WELL INSTALLATION WORK PLAN**



February 8, 2013

Ms. Gretchen Wagenseller  
Project Manager  
Arizona Department of Environmental Quality  
400 West Congress, Suite 433  
Tucson, Arizona 85701

Re: Work Plan 2013 Monitor Well Installation  
Miracle Mile WQARF Site  
URS Job No. 24097087.06000

Dear Ms. Wagenseller:

Enclosed for your review is the draft Work Plan for Monitor Well Installation at the Miracle Mile Site. This document is prepared by URS for the Arizona Department of Environmental Quality (ADEQ) as a part of Task Assignment Number 11-0068, in accordance with ADEQ Contract Number EV09-0100. This Work Plan outlines the process URS will use to install four new monitoring wells. If you have any questions concerning the Work Plan, please call me at 887-1800.

Sincerely,

URS Corporation

A handwritten signature in black ink that reads "William J. Neese".

William J. Neese, P.E.  
Project Manager

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**WORK PLAN  
2013 MONITOR WELL  
INSTALLATION  
MIRACLE MILE WQARF SITE  
TUCSON, ARIZONA**

**Prepared for  
ARIZONA DEPARTMENT OF  
ENVIRONMENTAL QUALITY**

**URS Job No. 24097087.04000  
February 2013**

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## **1.0 INTRODUCTION**

This Work Plan describes the overall scope of work and specific field activities that will be used during the installation of four wells at the Miracle Mile Water Quality Assurance Revolving Fund (WQARF) Site in Tucson, Arizona (Figure 1).

Contents of this document are based on current knowledge of the site and anticipated field conditions; however, slight changes may be necessary to meet actual conditions. These conditions and changes will be communicated to the Arizona Department of Environmental Quality (ADEQ) project manager prior to their implementation.

### **1.1 SITE BACKGROUND**

Based on the results of previous and current field investigations, trichlorethene is the predominant VOC within the Miracle Mile WQARF Site. A shallow, localized, perched groundwater zone was identified as a possible source to the regional aquifer, based on reports from historical boring and well completion logs.

Since January 1992, ADEQ has been routinely collecting groundwater level measurements and groundwater quality samples from the Miracle Mile WQARF Site. In addition to defining the extent of the plume and its migration through time, evaluation of the quarterly water level measurements and groundwater quality data provide information that assist in determining the locations of future monitor wells. All IRA monitor well locations are shown on Figure 2.

### **1.2 PROJECT CONTACT LIST**

Table 1 presents a list of telephone and fax numbers for personnel involved in this project. A list of emergency telephone numbers is provided in the site-specific Health and Safety Plan (HASP).

### **1.3 SCOPE OF WORK SUMMARY**

Well installation will consist of the mobilization of equipment to drill, install and develop wells at four locations shown on Figure 1.

There will be two medium regional wells drilled to a depth of 250 feet below ground surface (bgs), a shallow well drilled to 100 feet bgs, and a deep regional well drilled to 400 feet bgs. The proposed depths are approximate and may be adjusted based on field conditions. The proposed new monitoring well locations and bases for their selection are as follows:

- Monitoring Well IRA-38 will be a medium regional well that will serve as a sentinel well located between the northern extent of detected VOCs and the Villa Capri public supply well.
- Monitoring Well IRA-39 will be a medium regional well that will help define groundwater quality to the northwest, located approximately equidistant from IRA-5, IRA-24, and IRA-25.
- Monitoring Well IRA-40 will be a perched well to assess potential VOC contamination near the Friedman Recycling Facility.
- Monitoring Well IRA-41 will be a deep regional well to better define vertical extent of potential contamination in the vicinity of IRA-14A.

Proposed well construction schematic diagrams are included in Appendix A. During well installation, field staff will log the continuous soil core produced from the drill cuttings, using the Soil Boring Log form in Appendix B.

## **2.0 MONITORING WELL INSTALLATION**

This section describes the procedures and equipment that will be used to construct the groundwater wells during drilling, installation and development. Wells will be drilled by Boart Longyear, Inc., using the roto sonic method.

### **2.1 HEALTH AND SAFETY PLAN**

A written Health and Safety Plan (HASP) is required for hazardous waste investigations according to the Occupational Safety and Health Administration, Code of Federal Regulations 1910.120(b). In accordance with the URS health and safety program, a site-specific HASP has been prepared to govern the fieldwork activities.

Activities will be conducted under the assumption that encountering contamination is a possibility. The main chemical hazard involves exposure to VOC-contaminated groundwater during well construction activities. The site-specific HASP will guide the conduct of all field activities, including drilling, installation and development of wells and management of investigation derived waste (IDW). The HASP will identify physical and chemical hazards, specify required personal protection and monitoring equipment, summarize action levels, and outline emergency procedures for the tasks conducted in the field.

### **2.2 WELL PERMITTING**

ADEQ will obtain the access agreement with the land owner, Pima County. Boart Longyear will prepare and submit the appropriate traffic plans and permit application for work in the Right of Way. Boart Longyear will prepare and submit the Notice of Intention to Drill, Deepen, or Modify Monitor/Piezometer/Environmental Well form to the Arizona Department of Water Resources (ADWR) and the fee for the installation of each well. ADEQ will provide copies of the access agreement documenting landowner approval to be filed by Boart Longyear with the Well Completion reports to ADWR.

### **2.3 WELL INSTALLATION**

Monitoring wells will be drilled using the roto sonic method. The roto sonic method uses rotation and vibration to drill into soil. Roto sonic drilling method has been selected because it reduces IDW and provides intact core samples for lithologic descriptions.

Field geologists or engineers from URS will provide oversight for all investigative well drilling and construction activities. All downhole drilling equipment will be steam-cleaned before drilling at each location. The well locations will be prepared by placing plastic sheets beneath the drilling rig to protect the ground surface from oil and hydraulic fluid.

The wellbores will be drilled using a nominal 9-inch diameter core barrel. Depending on the soils encountered, a step-down to an 8-inch diameter drive casing may be necessary to complete the boring. Drill core and loose cuttings generated during drilling will be collected and transferred to roll-off bins. Water produced during the drilling and well development processes will also be contained and transferred in a trailer mounted tank to a temporary storage tank.

The boring will be logged from the surface to total depth. Information to be collected during drilling includes lithology, soil parameters, and occurrence of groundwater. Information collected during drilling will be recorded on the Soil Boring Log form included in Appendix B.

## **2.4 CORING AND SOIL SAMPLING PROCEDURES**

The driller will collect continuous core samples labeled with the corresponding base of the sample depth interval. The core shall be laid out in a sample storage area on a waterproof tarp or ground cover for each sampled interval in descending order. The storage area must allow for samples to be maintained in sequence and unmixed with surface material or other samples until they have been examined and logged by URS staff.

## **2.5 EQUIPMENT DECONTAMINATION**

All downhole equipment will be decontaminated between wells at a specified area at each location. Equipment will be steam cleaned or cleaned in an Alconox solution, triple rinsed, and allowed to air dry.

## **2.6 WELL CONSTRUCTION**

Schematic well construction diagrams are presented in Appendix A. The wells will be constructed using 4-inch nominal diameter PVC flush-threaded casing and screen. Total depth and screen interval may be adjusted based on field logging if a noticeable formation change occurs. Any portion of the borehole from total depth to the bottom of the casing will be sealed by backfilling with bentonite pellets before well construction should the well depth require reduction. The well will be constructed using screen with 0.020-inch slot openings. All screen and casing will be inspected in the field before placement in the well to ensure that each section

is clean and undamaged. The length of each section will be measured and recorded in the field notes.

After advancing the borehole to total depth and confirming the static water level by sounding, the screen and casing will be installed by threading each section of casing and screened casing together and lowering the partially-assembled screen/casing into the borehole. When the entire screen and casing has been assembled it will be suspended in the boring at the selected depths.

Annular materials will be installed after the casing has been placed. Sections of the drive casing will be removed as the annular space fills with material. The depth to the top of each material type will be verified by "tagging" the top of each material with a weighted tape measure.

Rounded 8-12 grade silica sand will be used to form the filter pack around the slotted casing. The filter pack will be vibrated into place around the screen using the roto-sonic drill motor to prevent bridging of the sand. The filter pack will be installed to a depth of at least 5 feet above the top of each screen to allow for settling during development. The filter pack material shall be free of shale, mica, clay, dirt, loam, organic impurities, and shall contain no iron or manganese in a form or quantity that will adversely affect the water quality. The filter pack material will be stored in bags and contained in a temporary storage area at the well site in such a manner as to prevent contamination. A two-foot thick layer of fine sand will be placed on top of the filter pack to prevent infiltration of the bentonite seal into the filter pack.

A 5-foot layer of sodium bentonite pellets or equivalent material will be placed above the fine sand and hydrated by adding water to the annular space between the casing and the borehole. The bentonite seal material shall contain no hazardous materials or gypsum. The bentonite grout seal will be allowed to hydrate for approximately 30 minutes before installing the rest of the annular seal.

Cement-bentonite grout will be tremied to create the annular seal from the top of the uppermost bentonite pellet seal to 5 feet below the ground surface. The Type II Portland cement grout seal material will consist of Portland cement slurry containing no more than 8 gallons of water per 94-pound bag of cement. The Portland cement will conform to ASTM Standard C150, Type II. The cement grout will consist of Portland cement with no greater than a 50 percent sand to cement ratio and up to 5 percent bentonite by volume. The upper 5 feet of the annulus will be filled with concrete during construction of the flush-mount traffic rated vault.

For the surface completion, the well will be completed with a water-tight cap and a protective steel, flush-mounted, traffic-rated well vault. The well vault will be placed within a concrete pad

level with surface grade. The vault lid and a tag inside the well vault will be labeled with the IRA-##, and the ADWR 55-#. All well construction information will be recorded on the Well Materials Tally Form shown in Appendix B.

## **2.7 FIELD DOCUMENTATION**

A field geologist or engineer will supervise the drilling and sampling activities. This individual will be responsible for maintaining the following documentation:

- Field observations and activities will be documented in a dedicated, bound log book used to record pertinent information that is not included on the other forms described below. Recorded information will include general Site conditions, daily weather, arrival and departure of subcontractors and visitors, equipment used at the Site, equipment problems, handling and disposal of produced water, departures from the field protocol and other relevant information;
- A soil boring log that records the drilling activities, describes soil conditions using the Unified Soil Classification System, as well as percent silt and clay, sand, and gravel;
- Health and Safety documentation as required by the HASP or Safe Work Plan.

## **2.8 SURVEYING**

New wells will be surveyed by an Arizona Registered Land Surveyor using Global Positioning Satellite (GPS) methods for horizontal coordinates accurate to 0.5 feet, and differential leveling methods for elevation accurate to +/- 0.02 feet, using site-compatible coordinate systems. Horizontal locations will be in Arizona State Plane Central Zone coordinates based on the NAD 83 Datum. Vertical locations will be in feet above mean sea level based on the NAVD 88 Datum.

### **3.0 WELL DEVELOPMENT**

Following construction, well development will be conducted using a service rig during a single mobilization. The wells will be developed to remove fine-grained sediments from the borehole wall and filter pack. The well development process is expected to take approximately 4 to 6 hours per well, depending on the condition of the well and local aquifer characteristics. Well development will consist of swabbing, bailing, and pumping. All well development equipment will be decontaminated, including surge block, bailer, piping, and temporary pump. All bailed sediments and bailed or pumped water will be contained for disposal as IDW.

#### **3.1 WELL DEVELOPMENT PROCEDURES**

Depth to water will be measured before the start of well development, following swabbing, and periodically during bailing and pumping operations. The well will be swabbed using a surge block. The well screen will be swabbed from the bottom to the top of the screen. After swabbing, sediment and turbid water will be removed from the bottom of the well via bailer.

Once the sediment and turbid water have been removed, a temporary development pump will be installed without a check valve. A 1-inch diameter sounding tube will be installed with a closed end installed on top of the pump assembly and perforated in the lower 10 feet with four ¼-inch diameter perforations per foot. The pump cable and sounder tube will be secured to the pump column with nylon ties, or stainless steel hose clamps. Tape will not be used other than as required for electrical splicing.

Pump development will proceed by pumping at the maximum sustainable rate and surging by shutting off the pump and allowing the column of water to fall into the well. Pump development will continue until water quality field parameters (pH, conductivity, and temperature) stabilize to within 10 percent for three measurements taken five minutes apart and turbidity has decreased to less than 10 nephelometric turbidity units (NTU). Water quality field data will be recorded on the Well Development form included in Appendix B.

#### **3.2 DEDICATED PUMPING EQUIPMENT**

Following well development, a dedicated pump capable of approximately 10 gallons per minute will be installed in the regional wells. The new pump will be decontaminated before installation. The pump will be installed with the pump intake in the center of the saturated screened interval. The pump cable and sounder tube will be secured to the pump column with nylon ties or stainless steel hose clamps.

Before beginning well evacuation, the static water level will first be measured to the nearest 0.01 foot using a decontaminated electric water level sounder. The well will then be pumped at a low flow rate of less than 25 gallons per minute (gpm) until three borehole volumes have been evacuated or until water quality field parameters have stabilized. Flow rate measurements will be recorded after each casing volume of water has been evacuated to confirm that the flow rate, and hence the evacuation time, is stable.

Water quality field parameters to be measured include pH, temperature, and specific electrical conductance. Multi-parameter instruments used to measure pH, temperature, and specific conductance will be calibrated on a daily basis before beginning well evacuations. Field measurements will be collected after each casing volume of water has been evacuated and will be considered to have stabilized when pH, conductivity, and temperature are within 10 percent for three measurements taken five minutes apart and turbidity has decreased to less than 10 NTUs.

## **4.0 INVESTIGATION DERIVED WASTE**

IDW will be handled, stored, treated and disposed of in accordance with federal, state, and local regulations. The governing regulations include 40 Code of Federal Regulations Parts 260-263, and Part 268. IDW generated during the well installation activities include drill cuttings soil and produced water from drilling and well development.

### **4.1 DRILL CUTTINGS**

Drill cuttings will be contained in soil sample bags or at the drill rig in a hopper mounted on a forklift. When full, the hopper will be dumped into a roll-off bin at the well site or a fenced storage area owned by Flowing Wells Irrigation District (FWID). The serial number for each bin will be noted in the field notes and on the composite sample identification. Each roll-off bin has a weight capacity of 9 tons, or approximately one-half the volume of the bin when filled with soil. Drill cuttings will be sampled, analyzed, and profiled prior to disposal. After waste authorization is received from the landfill, the bins will be transported to Butterfield Station Landfill.

#### **4.1.1 Drill Cutting Sampling**

One composite sample of drill cuttings will be collected from roll-off bins and submitted for analysis for the following parameters:

- Resource Conservation and Recovery Act Metals and zinc by EPA Methods 6010B/6020B/7471A using the Toxicity Characteristic Leaching Procedure EPA Method 1311.
- VOCs by EPA Method 8260B at the lowest detection limit available.

### **4.2 PRODUCED WATER**

Produced water will be generated during drilling of the saturated zone and well development. Produced water will be discharged to a trailer mounted tank and transferred to a storage tank at the fenced storage yard owned by FWID. When the tank is full, one water sample will be collected from the tank and submitted to the laboratory for analysis. Upon approval, the water will be transported by Busy D Pumping for disposal at their licensed facility.

#### 4.2.1 Produced Water Sampling

Produced water transferred to a storage tank will be sampled before disposal. A sample from the tank will be submitted for analysis for the following parameters:

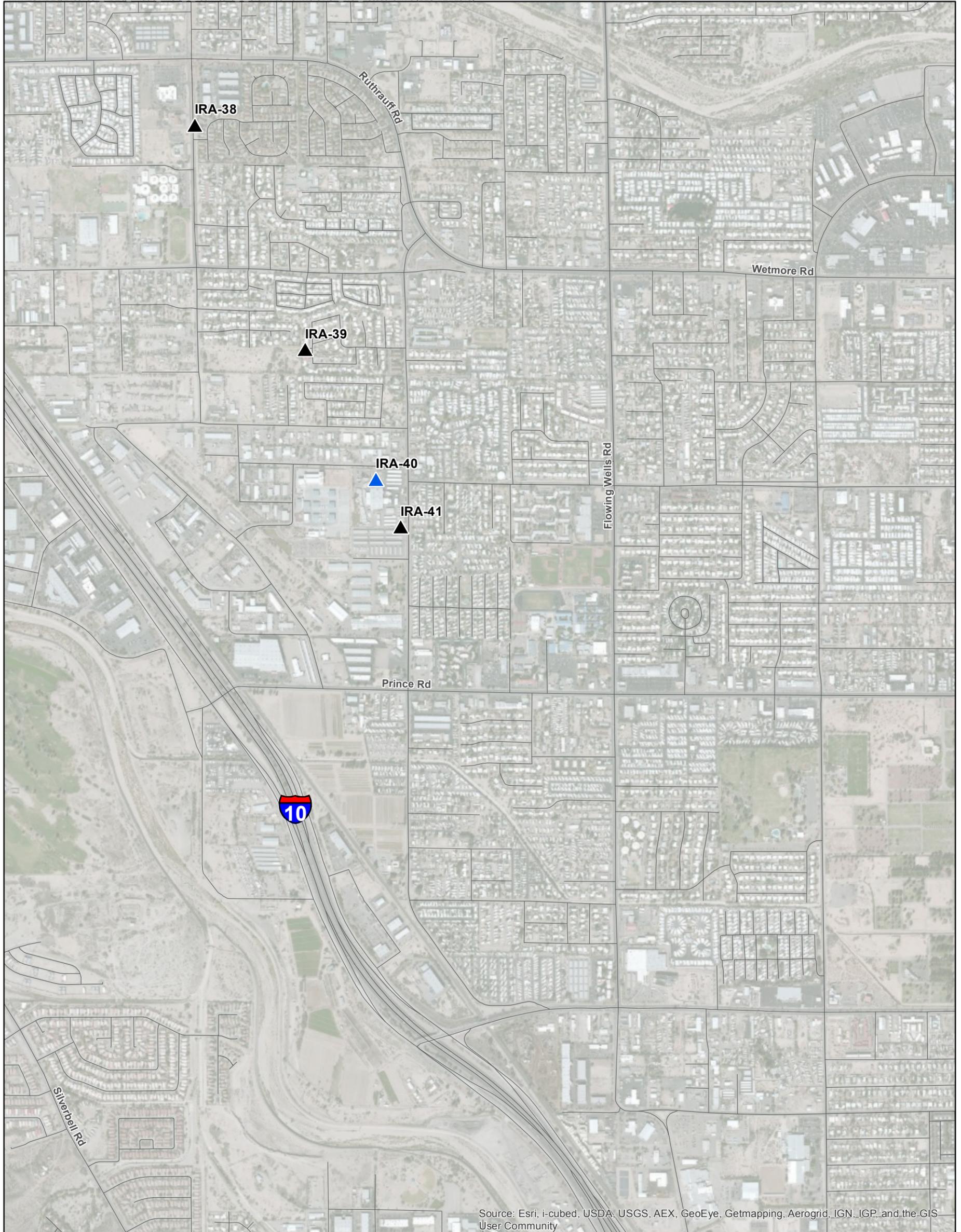
- Volatile Organic Compounds by EPA Method 8260B
- pH
- Flashpoint
- Oil & Grease
- 8 RCRA Metals
- Antimony
- Copper
- Molybdenum
- Zinc
- Total Suspended Solids
- Cobalt
- Tin
- Chemical Oxygen Demand

## **TABLES**

**Table 1**  
**Miracle Mile WQARF Site**  
**2013 Monitor Well Installation**  
**Contact List**

<b>Contact</b>	<b>Phone Number</b>	<b>Role</b>	<b>Responsibility</b>
<b>ADEQ</b>			
Gretchen Wagenseller	O: 520-628-6708 F: 520-628-6745	Client	ADEQ Contact
<b>Accutest Laboratories</b>			
Beth Proffitt	O: 602-501-5673 F: 408-588-0212	Lab Contractor	Analyze collected samples and report test results to URS
<b>HydroGeophysics, Inc</b>			
Chris Baldyga	O: 520-647-3315 M: 520-906-1054	Private Utility Locator Service	Locate and mark any subsurface obstructions not tagged by BlueStake
<b>URS Corporation</b>			
Will Neese	O: 520-487-2825 C: 520-465-7219	URS Project Manager(PM)	Overall responsibility for delivering the investigation on budget, on schedule and with a high degree of technical quality.
Adam Kneeling	O: 602-861-7429 C: 602-617-8707	Field Team Lead (FTL)	Coordinate and manage drill rig crews, gather all field data, and represent URS in client meetings.
Miles Hearn	O: 602-648-2509 C: 928-221-0454	Field Team Member	Oversee drilling activities and gather all necessary field data per borehole.
Derrick Maurer	O: 602-861-7469 C: 480-280-1840	Field Team Member	Oversee drilling activities and gather all necessary field data per borehole.
<b>Boart Longyear</b>			
Main Phoenix Office	O: 623-486-1881	Drill Contractor	Perform all tasks related to drilling and installation of monitoring wells, waste containerization, etc.
Driller	TBD		
<b>Busy D</b>			
BJ Faulk	O: 520-751-7765	Waste Pumping	Disposal of Waste Water

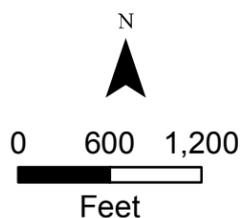
## **FIGURES**



Source: Esri, i-cubed, USDA, USGS, AEX, GeoEye, Getmapping, Aerogrid, IGN, IGP, and the GIS User Community

### Legend

- ▲ Proposed New Regional Monitor Well
- ▲ Proposed New Perched Monitor Well
- Road

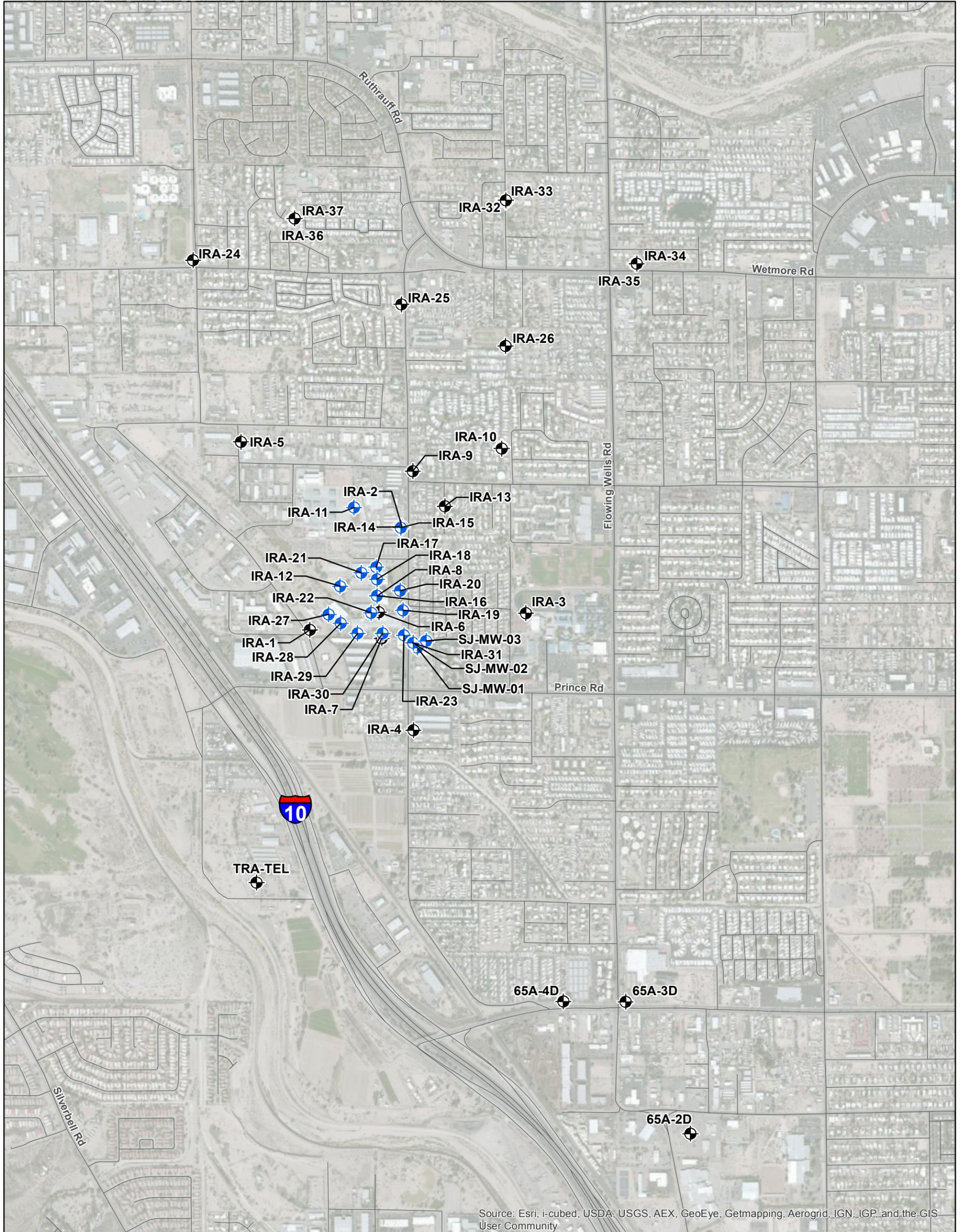


**Figure 1**  
**New Well Vicinity Map**  
**Miracle Mile WQARF Site**  
**Tucson, Arizona**

*DRAFT For Review Only*  
1/24/2013

Note:  
All proposed well locations are approximate and  
may be adjusted based on field conditions.

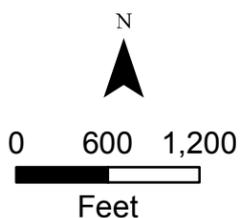




Source: Esri, i-cubed, USDA, USGS, AEX, GeoEye, Getmapping, Aerogrid, IGN, IGP, and the GIS User Community

### Legend

- Regional Monitor Well
- Perched Monitor Well
- Road



**Figure 2**  
**Site Monitoring**  
**Well Network Map**  
**Miracle Mile WQARF Site**  
**Tucson, Arizona**

**APPENDIX A**

**PROPOSED MONITOR WELL CONSTRUCTION DIAGRAMS**



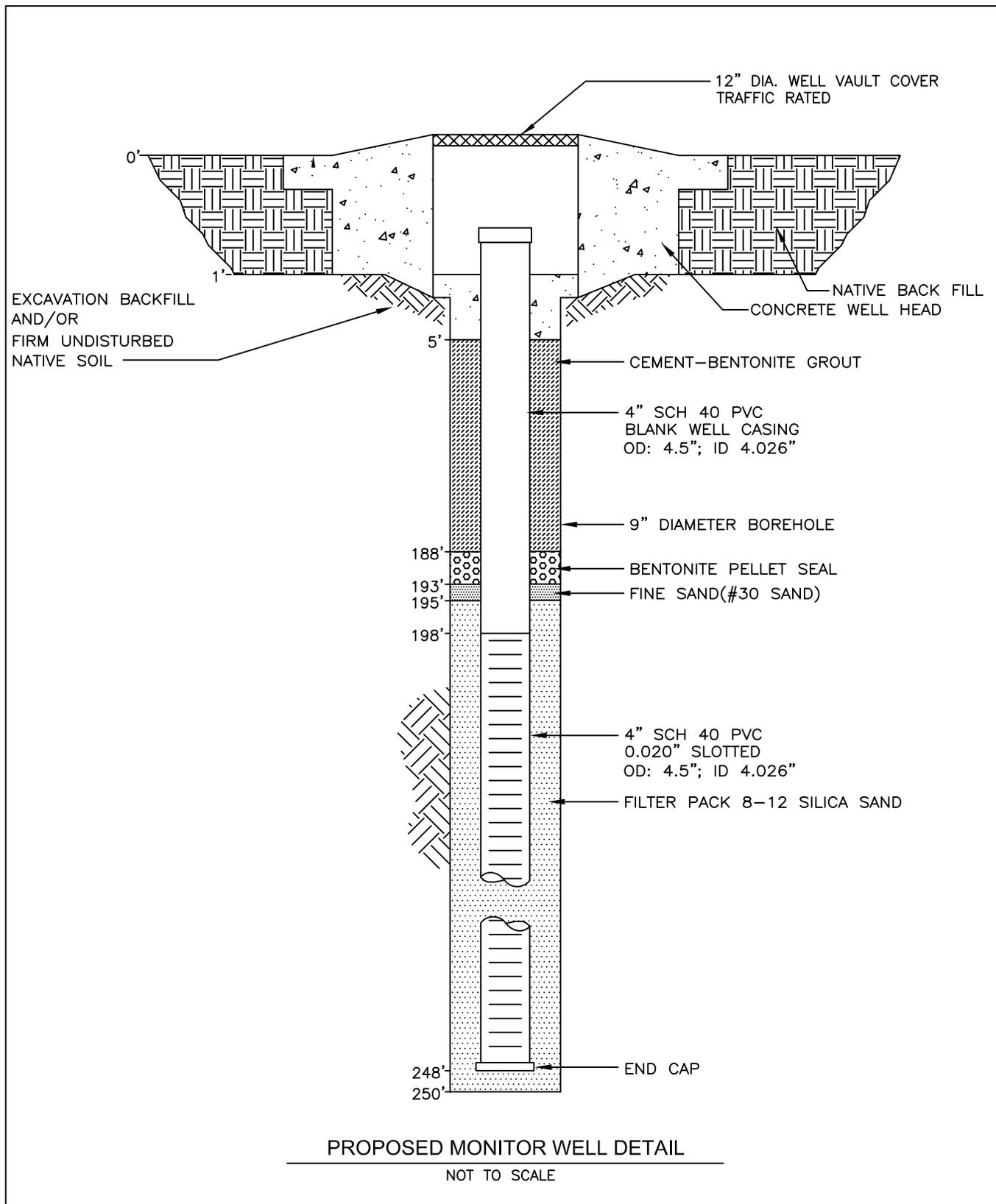
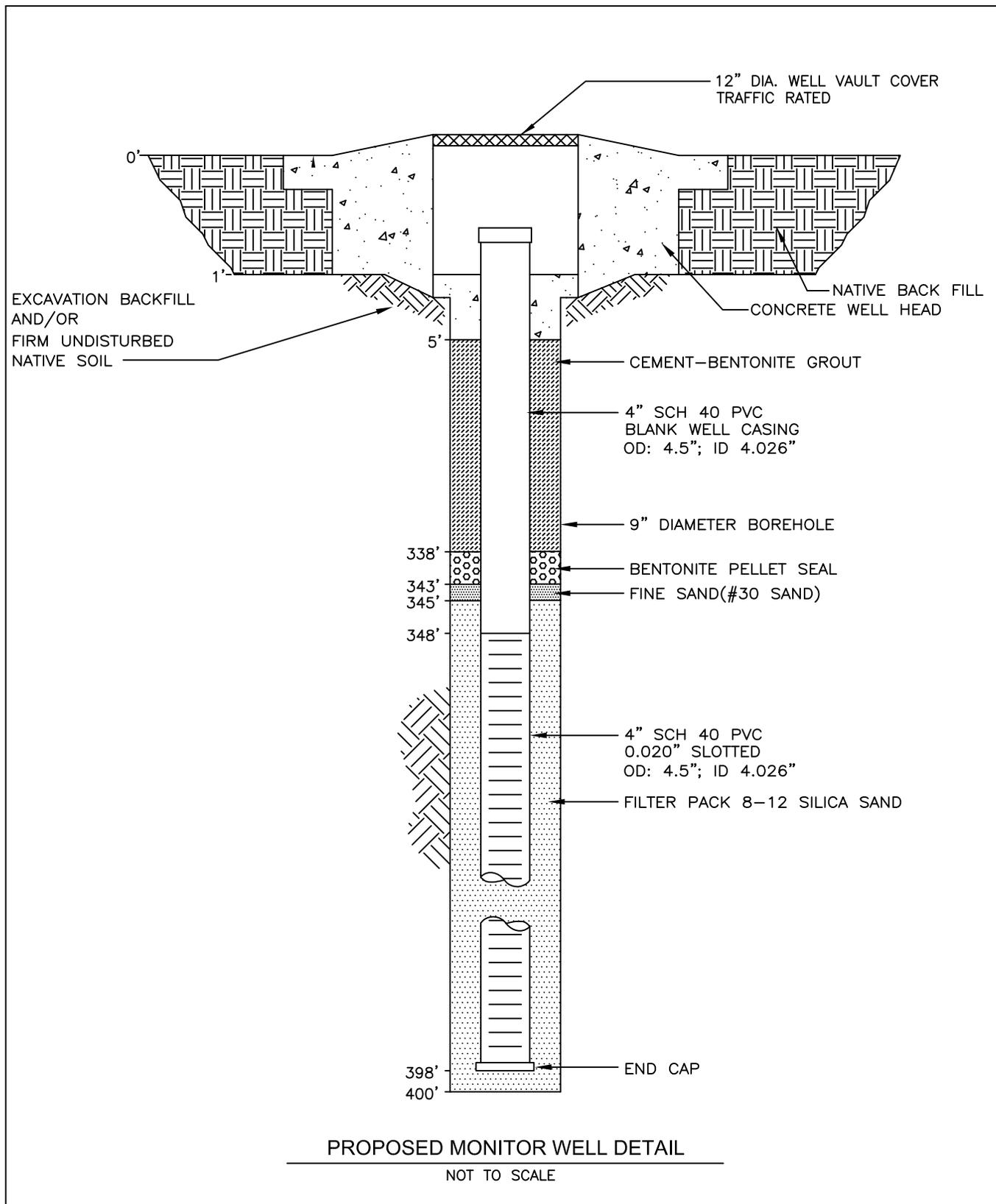


Figure 2  
Proposed Regional Intermediate Monitor Well Construction

Miracle Mile WQARF Site  
Tucson, Arizona





**Figure 3**  
**Proposed Regional Deep Monitor Well Construction**  
 Miracle Mile WQARF Site  
 Tucson, Arizona



**APPENDIX B**  
**FIELD FORMS**







**APPENDIX B**

**MIRACLE MILE WQARF SITE**

**SPRING JOINT SPECIALISTS CHROMIUM CONTAMINATED SOIL  
CHARACTERIZATION WORK PLAN**



February 6, 2013

Ms. Gretchen Wagenseller  
Project Manager  
Arizona Department of Environmental Quality  
400 West Congress, Suite 433  
Tucson, Arizona 85701

Re: Work Plan with Quality Assurance Project Plan for former Spring Joint Specialists Facility  
Miracle Mile WQARF Site  
Additional Soil Borings to Assess Chromium Contamination  
URS Job No. 24097087

Dear Ms. Wagenseller:

Enclosed for your review is the revised draft Spring Joint Specialists Chromium Contaminated Soil Characterization Work Plan. This document describes sampling of the soil at nine boring locations and is being prepared by URS for the Arizona Department of Environmental Quality (ADEQ) as a part of Task Assignment No. EV11-0068.

This Work Plan outlines the procedures that will be followed during the soil investigation to further assess the extent of the chromium contamination at the Site. In addition, a Quality Assurance Project Plan is attached that specifies the quality assurance and quality control measures that will be implemented. If you have any questions concerning this document, please call myself at 520-407-2825 or Jean Bierwirth at 520-407-2839.

Sincerely,

URS Corporation

William J. Neese, PE  
Project Manager

A vertical orange bar is located to the left of the title text.

**SPRING JOINT SPECIALISTS  
CHROMIUM CONTAMINATED  
SOIL CHARACTERIZATION  
WORK PLAN  
MIRACLE MILE WQARF SITE**

**Prepared for  
ARIZONA DEPARTMENT OF  
ENVIRONMENTAL QUALITY**

**URS Job No. 24097087  
February 2013**



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- 1 Spring Joint Site Proposed Boring Locations
- 2 Spring Joint Site Historic Soil and Groundwater Data
- 3 Spring Joint Site Shallow Soils Sampling Results

**APPENDIX**

- A Quality Assurance Project Plan, Soil Sampling at State of Arizona Miracle Mile Water Quality Assurance Revolving Fund Site



## **1.0 INTRODUCTION**

This Work Plan describes the overall scope of work and specific field procedures that will be utilized during the additional soil sampling at the former Spring Joint Specialties (Spring Joint) facility. The Spring Joint site is located within the boundaries of the State of Arizona Miracle Mile Water Quality Assurance Revolving Fund (WQARF) Site in Tucson, Arizona. The contents of this document are based on current knowledge of the site and anticipated field conditions; however, slight modifications may be required in order to meet actual field conditions. These conditions and changes will be communicated to the Arizona Department of Environmental Quality (ADEQ) project manager before implementation.

### **1.1 SPRING JOINT ADDITIONAL SOIL INVESTIGATION SUMMARY**

Additional sampling of the subsurface soil is proposed at the former Spring Joint Facility to better assess the nature and extent of total and hexavalent chromium in the soils. URS will drill nine soil borings to a total depth of approximately 100 feet below ground surface (bgs), or to the clay aquitard. Proposed boring locations are shown in Figure 1, and were determined based on information from previous investigations.

Drilling will be completed using a rotasonic drilling method with continuous soil cores collected and logged. Soil samples will be collected every 5 feet and analyzed for hexavalent chromium, for a total of 180 samples. In addition to sampling the soil for hexavalent chromium, samples of the clay aquitard will be analyzed for iron and manganese for characterization of the aquitard.

A report will be prepared summarizing the drilling of the borings, soil sampling, and analysis. Results will be summarized, soil-boring logs prepared, and an assessment of the nature and extent of the chromium contamination will be presented. A Quality Assurance Project Plan (QAPP) and a Health and Safety Plan (HASP) have been developed for the proposed work at Spring Joint. If access agreements cannot be obtained from property owners to complete the work in the FY 2013, the field work and reporting will be completed in FY 2014.



## 2.0 SITE BACKGROUND

### 2.1 SITE LOCATION AND BACKGROUND

The Spring Joint property is located at 3660 North Romero Road, in Tucson, Arizona. The property is currently vacant, but formerly contained a chromium plating operation. The site is located within the boundaries of the Miracle Mile WQARF area.

### 2.2 SITE SPECIFIC HYDROGEOLOGY

Hydrogeological units beneath the Miracle Mile WQARF Site primarily consist of basin-fill sediments, terrace deposits, and recent alluvium (clay, silt, sand, and gravel). A thin, discontinuous dark brown to red, moist clay layer occurs at depths of approximately 70 feet to 100 feet bgs, acting as an aquitard to inhibit the downward migration of water and creating perched groundwater zones. The regional aquifer occurs at depths of 155 bgs to 187 feet bgs with corresponding groundwater elevations of 2,128 to 2,122 feet above mean sea level. The groundwater flow direction is currently to the northwest, but has varied from the northeast to north to northwest. Groundwater flow direction as of October 2012 was to the north-northwest.

### 2.3 WELLS ON THE SPRING JOINT PROPERTY

In 2004, the following four well locations were drilled on the Spring Joint Property.

- **SJ-MW-1**

This well was drilled to a total depth of approximately 70 feet bgs and is a shallow, perched groundwater well. Lithographic logs from well installation describe yellow-stained soil at depths ranging from 25 to 61 feet bgs. During the most recent sampling event when access to the property was granted, this well could not be located.

- **SJ-MW-2**

This well, drilled to a total depth of approximately 74 feet bgs, is a shallow, perched groundwater well. This is the only shallow monitoring well on the Spring Joint property that consistently contains water.

- **SJ-MW-3**

This well was drilled to a total depth of approximately 94 feet bgs and is a shallow, perched groundwater well. During the most recent sampling event when access to the property was granted, this well could not be located.



- **IRA-31**

This well was drilled to a total depth of approximately 210 feet bgs into the regional aquifer. Drilling logs recorded during well installation described “yellow water” at 86 feet bgs. The static water level in this well is approximately 170 feet bgs. The well is located directly west of SJ-MW-2.

The levels of chromium found in the Spring Joint monitor wells may indicate a potential source of chromium found in the regional aquifer. However, chromium in monitor well IRA-31, completed in the regional aquifer at the Spring Joint Facility directly west of SJ-MW-2, does not show levels similar to those found in the shallow monitor wells. This may indicate that a means of transport from the perched groundwater zone to the regional aquifer does not currently exist at this well location. The locations and analytical results of the monitoring wells are presented on Figure 2.



### 3.0 PREVIOUS SOIL INVESTIGATIONS

#### 3.1 SOIL REMEDIATION LEVELS

The ADEQ soil remediation levels for chromium are given in the table below.

3.1.1 TABLE 1: Soil Remediation Levels for Chromium

	Residential Soil Remediation	Non-Residential Soil
Trivalent Chromium, (Cr III)	120,000 mg/Kg	1,000,000 mg/Kg
Hexavalent Chromium, (Cr VI)	30 mg/Kg	65 mg/Kg

Source: ADEQ, 2007

mg/Kg = milligrams per kilogram

#### 3.2 WESTERN TECHNOLOGIES, INC

In January 2004, Western Technologies, Inc., drilled a total of five borings at the Spring Joint property. Three of the soil borings were converted to shallow, perched groundwater monitoring wells, SJ-MW-1, SJ-MW-2 and SJ-MW-3, as described in Section 2.0. The remaining two soil borings, B1 and B2, were plugged and abandoned.

Soil samples collected from SJ-MW-1, B1 and B2 were analyzed for total chromium, trivalent chromium, and hexavalent chromium. The samples were taken at five-foot intervals beginning at the bottom of the 10 to 15 bgs interval to the maximum depth of the boring. The analytical laboratory reported hexavalent chromium soil concentrations above the Residential Soil Remediation of 30 mg/Kg in soil samples collected from boring SJ-MW-1 and B1. No samples exceeded the Residential Soil Remediation in boring B2. In addition to the soil samples, groundwater samples were collected from SJ-MW-1 and B2. Laboratory results showed total chromium of 18 milligrams per liter (mg/L) and 120 mg/L, respectively. Hexavalent chromium results were 9.90 mg/L and 120 mg/L, respectively. Figure 2 summarizes the groundwater and soil chromium data from previous sampling events.



### **3.3 URS CORPORATION**

During the period of June 2004 through February 2005, URS Corporation drilled 27 shallow soil borings, 6 indoor soil borings and 1 regional aquifer groundwater monitor well at the Spring Joint site to further characterize the horizontal and vertical extent of the chromium contamination.

The 27 shallow soil borings located throughout the property were sampled at a 0 to 6 inch depth and a 24 to 36 inch depth, for a total of 54 samples. All 54 samples returned detectable levels of chromium, ranging from 5.2 milligrams per kilogram (mg/Kg) to 2,800 mg/Kg. Twenty-seven of the 54 samples were analyzed for hexavalent chromium. Eleven of these samples had detectable levels of hexavalent chromium, ranging from 0.51 mg/Kg to 14 mg/Kg. The results of the shallow soil sampling are illustrated on Figure 3.



## **4.0 SCOPE OF WORK**

The scope of work for this project is summarized by the following tasks:

### **Task 1: Compile Information and Select Proposed Soil Boring Locations**

ADEQ and URS staff members have selected nine proposed boring locations as shown on Figure 1. The locations are based upon results from previous investigations at the Spring Joint site and are intended to further assess the nature and extent of the chromium contamination.

### **Task 2: Prepare the Sampling and Analysis Plan , Quality Assurance Project Plan and Health and Safety Plan**

The Sampling and Analysis Plan (SAP), QAPP and HASP have been prepared for this project. The SAP is presented in Section 6.0 of this work plan and the QAPP is in Appendix A. The HASP is site-wide and reviewed annually by the URS Health and Safety Officer. An overview of the HASP is provided in Section 7.0 of this Work Plan.

### **Task 3: Field Activities**

This task includes drilling nine borings, logging the boreholes, sampling the soil in accordance with the SAP, and managing the investigation-derived waste (IDW). All field activities will take place in accordance to the HASP. Photographs will be taken during field activities to provide a pictorial record of the subsurface soils. URS anticipates that this activity will take approximately six weeks to complete once drilling starts.

### **Task 4: Written Report**

Report writing will involve verification of the laboratory data and preparation of the final report detailing the field activities and analytical results. URS will work to complete the final report within four weeks of receiving all the laboratory results.



## 5.0 PROPOSED SOIL BORING LOCATIONS

### 5.1 SOIL BORING LOCATIONS

The current scope of work consists of boring nine holes to a depth of approximately 100 feet bgs at the Spring Joint facility to further assess the nature and extent of the chromium contamination. It is anticipated that the borings will also help better define the location and depth of the perched groundwater in the area. Additionally, the investigation will characterize the clay aquitard, better defining the extent and chemistry of the material. The proposed borings locations are B3 through B11 shown on Figure 1. These proposed locations are approximate and are subject to change based on physical access restrictions and field conditions.

### 5.2 SOIL BORING LOCATION RATIONALE

Previous soil investigations showed chromium contamination at several depths as illustrated on Figures 2 and 3. The proposed boring locations, illustrated on Figure 1, were selected to either confirm the results from previous investigations or to assess the extent of chromium contamination. Justification for each location is as follows:

#### *Proposed locations:*

- B3 - Located near the northwest corner of the adjacent parcel to the south, approximately 30 feet southwest of B1.  
Objective: To determine extent of contamination to the southwest of the former chrome plating tanks.  
Basis: In January 2004, Western Technologies, Inc. sampled B1 (illustrated on Figure 2) to a depth of 100 feet bgs and hexavalent chromium levels ranged from non-detect to 208 mg/Kg at 60 feet bgs.
- B4 - Located in the immediate vicinity of B1.  
Objective: To confirm results from Western Technologies, Inc. investigation in 2004.  
Basis: Results from the 2004 investigation indicate levels of hexavalent chromium ranging from non-detect to 208 mg/Kg at 60 feet bgs in samples taken from B1.



- B5 - Located southeast of B1.  
Objective: To delineate contamination to the southeast.  
Basis: Results from the 2004 investigation indicate levels of hexavalent chromium ranging from non-detect to 208 mg/Kg at 60 feet bgs in samples taken from B1. Note that currently the proposed location appears obstructed by a building. The final location will be field located depending on accessibility.
- B6 - Located in the immediate vicinity of the former chrome plating tanks.  
Objective: To determine levels of hexavalent chromium concentrations at the source area.  
Basis: No data at depths greater than 10 feet bgs are available in this area. Values of hexavalent chromium from samples taken at depths from 1 foot to 10 feet bgs ranged from 2.00 to 3,420 mg/Kg. Samples from greater depths are warranted to determine vertical extent of elevated hexavalent chromium levels.
- B7 - Located approximately 30 feet north of the former chrome plating tanks.  
Objective: To determine hexavalent chromium levels to the north of the source area.  
Basis: The extent of contamination at depths greater than 20 feet bgs has not been delineated in the area directly north of the former chrome plating tanks.
- B8 - Located approximately 60 feet northeast of the former chrome plating tanks.  
Objective: To determine hexavalent chromium levels to the northeast of the source area.  
Basis: Chromium contamination has been delineated in the area north-northwest of the former chrome plating tanks based on review of soil data from the IRA-31 monitoring well installation. However, there are no hexavalent chromium data from soils available at depths greater than 20 feet bgs from the area to the northeast.
- B9 - Located in the vicinity of former wash water underground storage tanks (USTs).  
Objective: Verify chromium detected in sludge from the USTs was contained by the USTs.  
Basis: Previous investigations when the former wash water USTs were removed, focused exclusively on hydrocarbon-based contamination. However, sludge from one of the USTs was sampled for total chromium and the result was 330 mg/Kg. Additionally, this location will assist in delineating extent of potential contamination to the east of the former chrome plating tanks.



B10 - To be centered approximately within the former “Unpaved Storage Yard”.

Objective: To confirm extent of contamination to the east-northeast.

Basis: No hexavalent chromium data from depths greater than 3 feet bgs have been recorded in this area. As illustrated on Figure 3, total chromium was reported at 1,500 mg/Kg in a sample collected from 0 to 6 inches bgs based on review of data presented in the *Spring Joint Facility Borings and Well Installation Report* (URS, June 2005). In addition, review of recommendations documented in the *Limited Site Characterization and Monitor Well Installation*, (Western Technologies, Inc., March 2004) indicates that this area was a potential concern.

B11 - To be located near the northeast corner of the property, in a location formerly labeled as “Drum Storage Area” and noted as having a trench with soil staining.

Objective: To assess contamination levels in the northeast quadrant.

Basis: No hexavalent chromium data at depths greater than 3 feet bgs have been recorded in this area. In addition, according to the Executive Summary of the *Phase I Environmental Site Assessment* (Western Technologies, Inc., December 2002), “The soil at the historical drum storage area, near the northeast corner of the property, and should be analyzed for chrome (*sic*), petroleum hydrocarbons, and solvents.” Sampling in this area was originally part of the scope of work for the *Limited Site Characterization and Monitor Well Installation*, (Western Technologies, Inc., March 2004) that was canceled mid-investigation by the owner of the Spring Joint facility. Further, total chromium was reported at 2,800 mg/Kg in a sample collected from 0 to 6 inches bgs based on review of data presented in the *Spring Joint Facility Borings and Well Installation Report* (URS, June 2005). There are overhead utility lines near this location and the borehole will be drilled a sufficient distance to ensure the safety of the field crew.



## **6.0 SAMPLING AND ANALYSIS PLAN**

### **6.1 SOIL SAMPLE COLLECTION**

#### **6.1.1 Procedure**

URS will collect soil samples in accordance with Accutest's Standard Operating Procedures (SOPs), Collection of Soil Samples for Metals Analysis and Collection of Soil Samples for Volatile Organic Compounds. The SOPs contain detailed instructions on handling of equipment and materials, decontamination and documentation (Appendix A of Soils QAPP).

A total of nine, 100-foot deep, borings will be drilled at the Spring Joint property. Soil samples will be collected every 5 feet, for a total of approximately 180 samples and submitted for analysis of hexavalent chromium. As discussed previously, a detailed lithographic log, as well as photographs, will be taken by URS field personnel to document the subsurface soil conditions.

#### **6.1.2 Sampling Containers, Labels, Handling, Preservation and Custody**

In the field, each sample container will be marked with the sampling location number, date, and time of sample collection. Sample containers will be securely packed in an ice filled cooler in preparation for delivery to the laboratory.

Upon receipt of the samples, the laboratory will notify the field manager if conditions or problems are identified that require immediate resolution. Such conditions include container breakage, missing or improper chain-of-custody (COC) forms, exceeded holding times and missing or improper sample labeling.

For each sample submitted to the analytical laboratory, an entry will be made on a COC form supplied by the laboratory. A minimum of one COC form will be completed for each day of sampling. The information recorded on the COC form includes the sampling date and time, sample identification number, requested analytes and methods, and sampler's name. The field supervisor will maintain custody of the samples until they are relinquished to the laboratory courier or shipping agent. A copy of the completed COC will be provided by the laboratory along with their final report of results.



## **6.2 FIELD DOCUMENTATION**

Sampling activities will be directed by a field supervisor who will be responsible for maintaining all field documentation. Sampling information will be recorded on COC forms and Daily Field Report forms. The documentation developed during field activities is as follows:

- A Daily Field Report form that records field activities and pertinent data, including general site conditions, daily weather, arrival and departure of subcontractors, equipment used onsite, equipment problems, sample ID's collected, handling and disposal of investigation derived waste, and other relevant information;
- Health and safety documentation as required by the HASP;
- The sample container labels and COC forms;
- A photograph log;
- A log describing and classifying the soil in each boring and indicating the samples submitted for analysis.

## **6.3 SOIL SAMPLE ANALYSES**

URS will submit soil samples to an Arizona licensed laboratory. Approximately 180 samples will be submitted for hexavalent chromium analyses. Samples will be analyzed for hexavalent chromium using EPA Method 7196A.

For the highest detected hexavalent concentrations, further analysis of the specific soil samples using Synthetic Precipitation Leaching Procedure (SPLP) (EPA Method 1312) and Total Chromium will be discussed with ADEQ staff. SPLP simulates the leaching of compounds left on the ground which are exposed to natural rainfall and will be used to calculate hexavalent and total chromium Groundwater Protection Levels. For the clay aquitard, further examination will include Manganese (III), Iron (II), sulfide/sulfate, Total Organic Carbon and Dissolved Oxygen. Testing in the clay aquitard will be used to quantify its capability to hold chromium and prevent movement into the regional aquifer. The data from this analysis could be useful for future remediation activities at the Spring Joint site.



## **6.4 GROUNDWATER SAMPLES**

It is possible that perched groundwater may be encountered during the drilling activities. If this occurs, groundwater will be sampled for total and dissolved chromium and hexavalent chromium. If groundwater samples are collected, they will be analyzed for total chromium using EPA Method 6010B, analyzed for dissolved chromium using EPA Method 6010B, and hexavalent chromium using EPA Method SM3500CrD. The dissolved chromium analysis requires field filtering. This additional information may assist in the overall remediation investigation of the Miracle Mile WQARF area.



## 7.0 HEALTH AND SAFETY OVERVIEW

A site-specific HASP has been prepared for the fieldwork at the Spring Joint facility. This section of the Work Plan is intended to serve only as an overview.

### 7.1 LEVELS OF PROTECTION

It is anticipated that all site activities will be performed in Level D personal protective equipment. This shall consist at a minimum of steel toe boots, hardhat, work gloves, safety glasses, and hearing protection. Should water or wet soil conditions be encountered, all personnel will be required to use at a minimum, disposable latex gloves under work gloves and possibly don coated overalls or respirators.

URS personnel will monitor total organic vapors with a calibrated photoionization detector (PID) in workers' breathing zone as drilling progresses, to ensure that there is no potential for exposure. The HASP has established a 10 parts per million (ppm) action levels. Communication and emergency information is as follows:

#### **Gretchen Wagenseller, ADEQ Project Manager**

Office Phone: (520) 628-6708

Cell Phone: (520) 240-8590

#### **Will Neese, URS Project Manager**

Office Phone: (520) 407-2825

Cell Phone: (520) 465-7219

<b>Emergency Phone Numbers -</b>		
<b>Organization</b>	<b>Name</b>	<b>Phone numbers</b>
Police	Tucson Police	<b>911</b>
Ambulance	Tucson Fire Department	<b>911</b>
Hospital	Northwest Medical Center	<b>911</b>



Fire/HAZMAT	Tucson Fire Department	<b>911</b>
Poison Control Center	--	<b>(800) 332-3073</b>
URS Occupational Health Manager	Jeanette Schrimsher, RN	<b>(866) 326-7321 (24/7)</b>
URS HSE Representative	Armando Jimenez	<b>(520) 906-9177</b>
URS Regional HSE Manager	Tim Joseph	<b>(303) 740-2767</b>

To reach the hospital from the intersection of Wetmore Road and Flowing Wells Roads, proceed north on Flowing Wells for three miles (Flowing Wells becomes La Canada after crossing the Rillito Wash. At River Road, turn left (west), and go approximately one mile to La Cholla Blvd. Head north on La Cholla Blvd., Northwest Medical Center will be on the right just before reaching Orange Grove; it is at the southeast corner of Orange Grove and La Cholla Blvd. at 6200 N. La Cholla Blvd., Tucson, Arizona.





## **8.0 FIELD ACTIVITIES**

### **8.1 PERMITTING AND AUTHORIZATION FOR DRILLING**

All public and private utilities will be located approximately one week before drilling at each location. URS will contact Arizona Blue Stake for location of public utilities, and a private utility location service for private utilities. Authorization and access to drill will be obtained by ADEQ from the respective property owners before commencing drilling activities.

### **8.2 SURVEYING**

The proposed locations of the borings will be surveyed before drilling activities. As-built locations will also be surveyed in the event that boring locations are moved due to actual site conditions.

### **8.3 DRILLING PROCEDURES**

The soil borings will be drilled using roto-sonic drilling method. The sonic drilling method uses a high frequency mechanical vibration and low speed rotational motion coupled with down pressure to advance the coring drill bit, followed by the drilling casing. The sonic rig has a hydraulically-powered drill head or oscillator, which generates adjustable high frequency vibrational forces. The sonic head is attached directly to the core barrel, drill pipe, or outer casing, transmitting the high frequency vibrations downward through the drill steel to the face of the drill bit to advance the bore hole. This creates displacement by fluidizing unconsolidated soil, or by shearing through consolidated soils and cobbles.

Continuous, 4-inch diameter soil cores will be obtained from each boring using a core barrel. The cores will be retrieved to the surface and vibrated out of the core barrel into a sample sleeve. Each core will be described by the URS site geologist. Drilling of the soil borings will be to a depth of approximately 100 feet bgs or to the clay aquitard.

### **8.4 LITHOLOGIC LOGGING**

The continuous soil samples collected during the drilling process will be contained in a sealed plastic sample sleeve. The full sample sleeve will be moved from the drill rig to a designated location for field logging by the site geologist. Before describing the lithology, a calibrated PID will be inserted into the sample sleeve at each 2.5-foot soil core interval and the total



measurement of organic vapors will be recorded on the lithologic log. Once the PID readings have been recorded, the sample sleeve will be cut open providing access to the soil core. The soil core will then be sliced in half making the inside of each core visible for logging. Each soil core will be characterized in accordance with the Unified Soil Classification System.

#### **8.4.1 Lithologic Log**

Soils will be logged in accordance with ASTM standards. The petrology will also be identified based upon the texture, fabric, and structure of the materials encountered.

The following information will be noted on the lithologic log:

- Type and percentage of various grain sizes;
- Angularity of coarse grained particles;
- Reaction with HCl;
- Consistency;
- Cementation;
- Plasticity;
- Moisture conditions; and
- Color using the Munsell charts.

#### **8.5 SOIL SAMPLING**

Soil samples will be collected every five feet, for an approximate total of 180 samples. The samples will be collected in a glass container and cooled to approximately 4° C for hexavalent chromium analysis. All samples will be marked for hold for possible future total chromium and SPLP analysis. All samples will be collected and delivered to the laboratory using COC procedures. The Sampling and Analysis Plan is included in Section 6.0 of this Work Plan.

#### **8.6 BORING CLOSURE**

The nine soil borings will be abandoned in compliance with Arizona Revised Statute Title 45, Chapter 2, Article 8 and pursuant to Arizona Administrative Code R12-15-816. They will be filled from total depth to the ground surface with cement/bentonite slurry. As-built locations will also be surveyed in the event that boring locations are moved due to actual site conditions.



## **8.7 INVESTIGATION DERIVED WASTE MANAGEMENT**

During the boring installations, URS will collect and carefully document PID readings from each sampling interval. Intervals indicating the presence of VOCs will be segregated based upon the measured concentrations. Soil intervals indicating concentrations less than the 2 ppm PID detection limit will be placed into a roll-off. Soil indicating detectable concentrations of VOCs or visible signs of chromium (yellowish-green color) will be temporarily placed into a lined 55-gallon drum, and transported to the staging area. The drums may then be transferred into a roll-off based upon the observed PID concentrations or analytical results.

Soil intervals with PID concentrations indicating 100 ppm or greater, or exhibiting yellowish-green color, will be sub-sampled for laboratory analysis of VOCs and chromium. Soil with detectable concentrations of VOCs or chromium will remain in the 55-gallon drums until project completion. The soil may eventually be transferred into a roll-off, and blended with similar soil.

Four-inch diameter soil cores will be obtained throughout the entire borings. The cores will be maintained at the central staging area until completion of the drilling program. Upon completion of drilling, the cores will be placed into the appropriate roll off container along with the soil cuttings to await profiling characterization. Profiling for soil disposal will include the collection of one composite soil sample from each roll off container. Each sample will be analyzed using TCLP methods for VOCs and RCRA metals.

It is possible that groundwater will be encountered during the drilling activities. This water will be pumped into the dewatering box to separate water from sediments. If necessary, the water will then be pumped/treated through the GAC system and stored in a 6,500-gallon Baker Tank until characterization.

The decontamination water will be maintained in a lined impoundment and allowed to evaporate throughout the duration of the project. Any remaining decontamination water will be pumped into the dewatering box. Water remaining at the end of drilling activities will be sampled and disposed in accordance with all applicable regulations. In summary, URS has identified four potential waste streams for this project.



### **Clean Soil**

Clean soil includes soil with no PID reading and for which a composite sample indicates no detectable concentrations of VOCs or chromium above detection limits. Clean soil will be transported to the Los Reales Landfill operated by the City of Tucson Solid Waste Department.

### **Solid Waste**

Solid waste includes discarded personal protective equipment and soil with detectable concentrations of VOCs or chromium, either by PID or laboratory analysis, which is not considered characteristically hazardous for VOCs or chromium. Solid waste will be transported to the Los Reales Landfill, for disposal as solid waste in their lined cell.

### **Hazardous Waste**

Hazardous waste includes soil that does not pass the TCLP methods. It is likely that some soils from the Spring Joint facility will exceed the TCLP limit for chromium. In the event that hazardous waste is identified, the soil will be transported to a RCRA-approved disposal facility in accordance with all applicable regulations.

### **Liquid Waste**

Liquid waste includes all of the liquid IDW generated during the drilling activities. Potential liquid sources include possible groundwater contacted above the perched layer and decontamination/wash water from drilling/sampling activities. A minimal amount of liquid waste is anticipated for this project; however, all liquids will be captured and stored in a portable tank. The tank will be sampled and the liquid disposed of, in accordance with all applicable regulations.



## **9.0 REFERENCES**

URS, 2005. Spring Joint Facility Borings and Well Installation Report, Miracle Mile WQARF Site Remedial Investigation. June 11, 2005.

Western Technologies, 2002. Phase I Environmental Site Assessment Commercial Property 3660 North Romero Road Tucson, Arizona: Prepared by Western Technologies, Inc. for the Spring Joint Specialists, Inc.

\_\_\_\_\_. 2004. Limited Site Characterization and Monitor Well Installation, Spring Joint Specialists, Inc., 3660 North Romero Road, Tucson, Arizona: Prepared by Western Technologies, Inc. for the Spring Joint Specialists, Inc, March 8, 2004.



**FIGURES**

Figure 1

### Spring Joint Site Proposed Boring Locations

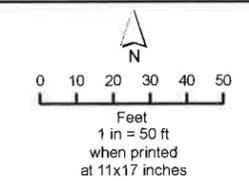
Miracle Mile WQARF Site Tucson Arizona

#### Legend

-  Proposed Boring
-  Spring Joint Property Boundary
-  Asphalt Cap Approximately 53 feet x 56 feet



Note:  
All sample locations are approximate and proposed boring locations may be adjusted based on field conditions.



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DRAFT: 2/5/2013  
**URS**



Figure 2

Spring Joint Site  
Historic Soil and  
Groundwater Data

Miracle Mile WQARF Site Tucson Arizona

Legend

- Perched Well
- Regional Monitor Well
- Approximate Western Tech Boring Location
- Approximate Boring Locations 1990
- Former Underground Storage Tank (UST)
- Approximate Western Tech Excavation Site\*
- Spring Joint Property Boundary

\*Following samples were collected from stockpiles generated during excavation outlined on this figure in the southwest quadrant of the property.

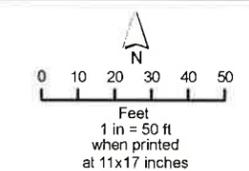
Location	Date	Total Cr	Cr(VI)	TCLP
SJ-50	8/15/2003	1700	NM	54 mg/kg
SJ-51	8/15/2003	8200	NM	320 mg/kg

Notes:  
TCLP: Toxicity Characteristic Leaching Procedure  
NA: Not Analyzed  
NM: Not Measured  
ND: Not Detected

- Groundwater Samples (in mg/l)
- Soil Samples (in mg/kg)

For analytical results associated with soils (includes sludge and stockpile materials):  
x.x Result below Residential Soil Remediation Level  
xx.x Result above Residential Soil Remediation Level  
xxx.x Result above Non-Residential Soil Remediation Level

For analytical results associated with groundwater:  
x.x Result below Arizona Water Quality Standard  
xx.x Result above Arizona Water Quality Standard



**IRA-31**

Date	Total Cr
12/2004:	0.040
04/2005:	0.053
10/2005:	0.020
04/2006:	0.018
10/2006:	0.017
04/2007:	NM
10/2007:	NM
04/2008:	NM
11/2008:	0.021
10/2010:	0.02
12/2011:	ND

**SJ-MW-02**

Date	Total Cr	Cr(VI)
04/2004:	NM	NM
05/2004:	NM	130
12/2004:	NM	NM
04/2005:	80	NM
10/2005:	NM	99
04/2006:	91	NM
10/2006:	74	NM
04/2007 - 4/2008:	NM	NM
11/2008:	83	NM
10/2010:	83.2	NM
12/2011:	NM	NM

**SJ-1**

8/31/1990  
Total Cr: 7.5  
13.0 to 16.0 Feet

**SJ-29-DS-BW**

5/7/2003  
Total Cr: ND  
Cr(VI): NS  
4' deep

**SJ-8**

2/18/2003  
Total Cr: 350  
Cr(VI): 27.8  
2' deep

**IRA-31**

6/11/2005

Depth	Total Cr	Cr(VI)
5 Feet	9.0	<0.5
10 Feet	ND	NA
15 Feet	6.3	<2.0
20 - 65 Feet	ND	NA
70 Feet	9.5	8.6
75 Feet	11	12
80 Feet	14	9.2
85 Feet	ND	NA
90 Feet	18	16
95 Feet	15	10
100 Feet	11	8.4
105 Feet	16	1.5
110 - 130 Feet	ND	NA
135 Feet	6.4	2.5
140 - 210 Feet	ND	NA

**SJ-14**

3/4/2003  
Total Cr: 860  
Cr(VI): 824  
"Shallow End", maybe 2' based on sample id SJ-14-N-2

**SJ-MW-03**

Date	Total Cr	Cr(VI)
04/2004:	NM	
05/2004 - 10/2005:	NM	
04/2006:	ND	
10/2006:	ND	
04/2007 - 10/2011:	NM	

**SJ-7**

2/18/2003  
Total Cr: 460  
Cr(VI): 12.9  
2' deep

**SJ-30-DS-BE**

5/7/2003  
Total Cr: ND  
Cr(VI): NS  
4' deep

**SJ-48**

8/15/2003  
Total Cr: 220  
Cr(VI): 78.4  
4' deep

**SJ-45**

8/15/2003  
Total Cr: 78  
Cr(VI): 33.8  
2' deep

**SJ-47**

8/15/2003  
Total Cr: 590  
Cr(VI): 510  
6' deep

**SJ-2**

8/31/1990  
Total Cr: 11.8  
12.0 to 14.0 Feet

**SJ-4**

8/31/1990  
Total Cr: 83.3  
11.0 to 14.04 Feet

**SJ-43**

8/15/2003  
Total Cr: 13  
Cr(VI): 17.5  
8' deep

**SJ-41**

8/15/2003  
Total Cr: 1100  
Cr(VI): 394  
10' deep

**SJ-3**

8/31/1990  
Total Cr: 13.2  
13.0 to 17.13 Feet

**WT Romero Sludge/H3161**

4/16/2003  
Total Cr: 330  
Cr(VI): NM

**B2**

1/21/2004

Depth	Total Cr	Cr(VI)
15 Feet	12.2	ND
20 Feet	5.75	ND
25 Feet	3.39	2.40
30 Feet	3.85	ND
35 Feet	4.55	2.00
40 Feet	4.55	2.60
45 Feet	3.45	ND
50 Feet	4	ND
55 Feet	8.41	2.20
60 Feet	3.37	ND
65 Feet	10.6	9.2
70 Feet	17	12.6

**SJ-5**

8/31/1990  
Total Cr: 10.5  
10.0 to 12.6 Feet

**B2**

Date	Total Cr	Cr(VI)
01/2004:	120	120

**B1**

1/20/2004

Depth	Total Cr	Cr(VI)
15 Feet	11.8	3.00
20 Feet	3.66	ND
25 Feet	3.7	ND
30 Feet	3.69	ND
35 Feet	4.27	ND
40 Feet	4.88	ND
45 Feet	4.62	ND
50 Feet	2.92	ND
55 Feet	5.52	ND
60 Feet	364	208
65 Feet	12.8	3.4
70 Feet	13.6	2.2
75 Feet	7.05	ND
80 Feet	1.57	ND
85 Feet	2.4	ND
90 Feet	4.47	ND
95 Feet	3.64	ND
100 Feet	12.3	ND

**SJ-15**

3/4/2003  
Total Cr: 780  
Cr(VI): 598  
Maybe 1' based on sample id SJ-15-S-1

**SJ-49**

8/15/2003  
Total Cr: 260  
Cr(VI): 191  
4' deep

**SJ-42**

8/15/2003  
Total Cr: 19000  
Cr(VI): 3420  
8' deep

**SJ-MW-01**

1/13/2004

Depth	Total Cr	Cr(VI)
15 Feet	8.09	2.00
20 Feet	6.6	6.6
25 Feet	18.9	20.6
30 Feet	37.2	7.2
35 Feet	133	73
40 Feet	80.1	41.6
45 Feet	46.4	27
50 Feet	242	143
55 Feet	222	159
60 Feet	129	87.2
65 Feet	22.1	14.2
70 Feet	56.8	36.2
72.5 Feet	2.73	2.00

**SJ-13**

3/4/2003  
Total Cr: 1400  
Cr(VI): 726  
"Deep End", maybe 10' based on sample id SJ-13-N-10

**SJ-46**

8/15/2003  
Total Cr: 25  
Cr(VI): 2.92  
4' deep

**SJ-44**

8/15/2003  
Total Cr: 730  
Cr(VI): 664  
5' deep

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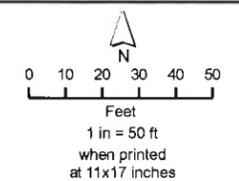
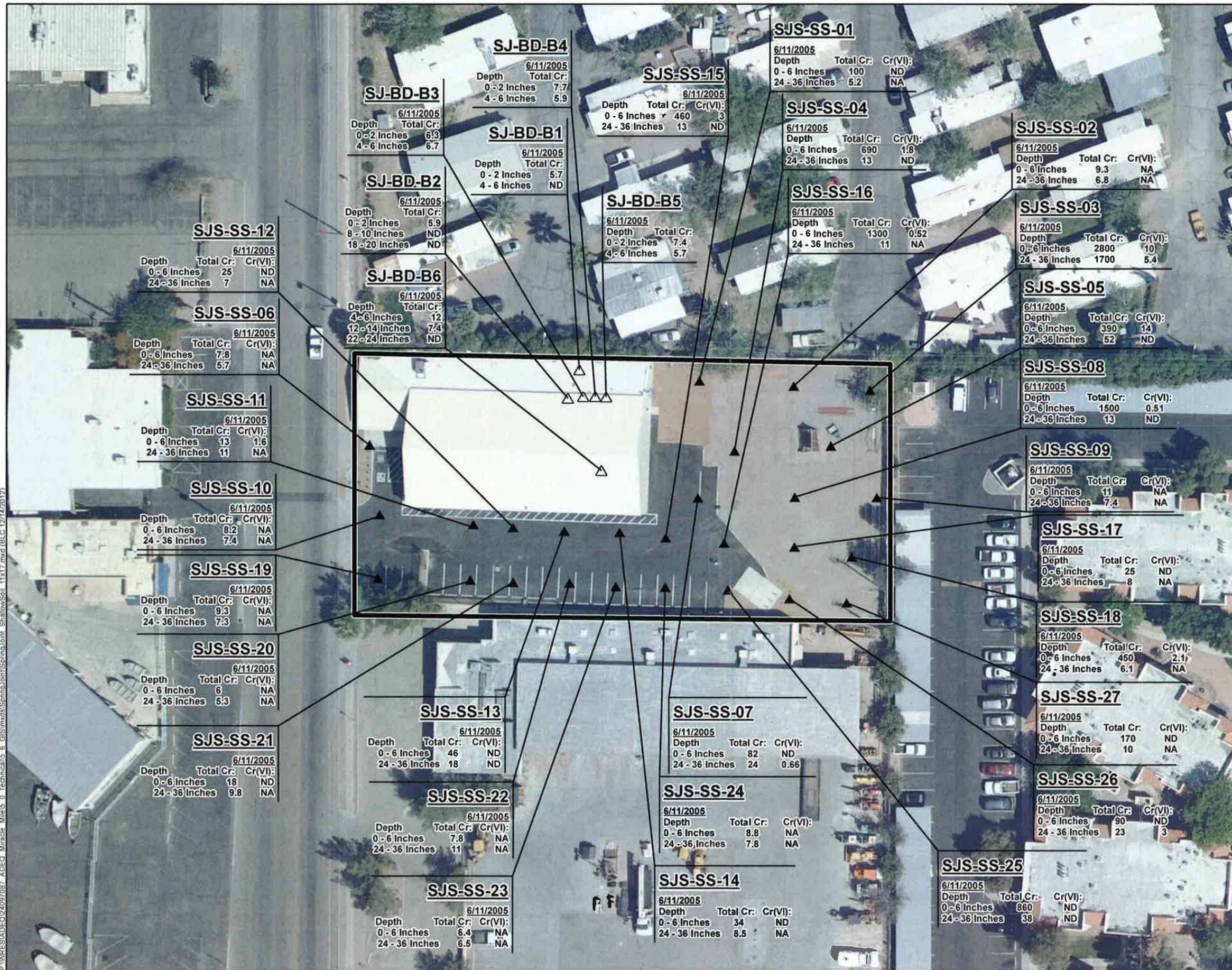
Figure 3  
Spring Joint Site  
Shallow Soils  
Sampling Results

Miracle Mile WQARF Site Tucson Arizona

Legend

- △ Interior 2005 Borings
- ▲ Shallow 2005 Borings
- ▭ Spring Joint Property Boundary

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**APPENDIX A**

**QUALITY ASSURANCE PROJECT PLAN**

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**QUALITY ASSURANCE PROJECT PLAN  
SOIL SAMPLING AT MIRACLE MILE  
WQARF SITE  
TUCSON, ARIZONA**

**Prepared for  
ARIZONA DEPARTMENT OF  
ENVIRONMENTAL QUALITY**

**Prepared by  
URS CORPORATION  
URS Job No. 24097087  
February 2013**

**APPROVAL SHEET AND DISTRIBUTION LIST  
QUALITY ASSURANCE PROJECT PLAN  
FOR SOIL SAMPLING AT MIRACLE MILE WQARF SITE  
FEBRUARY 2013**

---

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(408) 588-0200

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Date



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## APPENDICES

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- B EPA/ADEQ POLICIES AND PROCEDURES
- C URS DATA VERIFICATION PROCEDURES

<b>LIST OF ACRONYMS</b>	
ADEQ	Arizona Department of Environmental Quality
ADHS	Arizona Department of Health Services
ASTM	American Society for Testing and Material
CCV	Continuing Calibration Verification
COC	Chain-of-custody
DQO	Data Quality Objective
EPA	U.S. Environmental Protection Agency
ID	Identification
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
mg/kg	milligrams per kilogram
MMIA	Miracle Mile Interchange Area
MS	Matrix Spike
MSD	Matrix Spike Duplicate
PID	Photoionization Detector
QAPP	Quality Assurance Project Plan
QA	Quality assurance
QC	Quality control
QSM	Quality System Manual
RES-SRL	residential Soil Remediation Levels
RI	Remedial Investigation
RPD	Relative Percent Difference
%R	Percent recovery
SRL	Soil Remediation Level
SOP	Standard Operating Procedure
URS	URS Corporation
UST	Underground Storage Tank
VOCs	Volatile Organic Compounds
WQARF	Water Quality Assurance Revolving Fund

## CROSS REFERENCE OF QUALITY ASSURANCE ELEMENTS

The following table contains a cross reference between this document and the required elements specified in U.S. Environmental Protection Agency Requirements for Quality Assurance Plans for Environmental Data Operations, Interim Final, EPA QA/R-5 (EPA, 2001). This cross reference is provided to assist the reader in determining where the required elements are addressed in this Quality Assurance Project Plan.

QUALITY ASSURANCE (QA)/R-5 ELEMENTS		COMMENT ADDRESSED IN THIS QAPP
A1	Title and Approval Sheet	QAPP Cover and Signature Page
	Title	QAPP Cover
	Organization's name	QAPP Cover
	Dated signature of project manager	QAPP Page i
	Dated signature of QA officer	QAPP Page i
	Other signatures, as needed	QAPP Page i
A2	Table of Contents	QAPP Page ii
A3	Distribution List	QAPP Page i
A4	Project/Task Organization	QAPP Section 2.0
	Identifies key individuals, with their responsibilities (data users, decision-makers, project QA manager, subcontractors, etc.)	QAPP Section 2.0
	Lines of authority and reporting responsibilities	QAPP Section 2.0
A5	Problem Definition/Background	QAPP Sections 1.0 and 4.0
	Clearly states problem or decision to be resolved	QAPP Section 4.0
	Provides historical and background information	QAPP Section 1.0
A6	Project/Task Description	QAPP Section 1.0 and 4.0
	Lists measurements to be made	QAPP Section 1.0 and 4.0
	Cites applicable technical, regulatory, or program-specific quality standards, criteria, or objectives	QAPP Section 4.0
	Notes special personnel or equipment requirements	Not Applicable
	Provides work schedule	QAPP Section 1.0
	Notes required project and QA records/reports	QAPP Section 8.0
A7	Quality Objectives and Criteria for Measurement Data	QAPP Section 4.0
	States project objectives and limits, both qualitatively and quantitatively	QAPP Section 4.0
	States and characterizes measurement quality objectives as to applicable action levels or criteria	QAPP Section 4.0
A8	Special Training/Certification	QAPP Section 3.0
	States how provided, documented, and ensured	QAPP Section 3.0
A9	Documents and Records	QAPP Section 8.0
	Lists information and records to be included in data report (e.g., raw data, field logs, results of Quality Control (QC) checks, problems encountered)	QAPP Section 8.0
	States requested lab turnaround time	QAPP Section 8.0
	Gives retention time and location for records and reports	QAPP Section 8.0
B1	Sampling Process Design (Experimental Design)	QAPP Section 5.0
	States the following:	
	Type and number of samples required	QAPP Section 4.1
	Sampling design and rationale	QAPP Section 4.1
	Sampling locations and frequency	QAPP Section 4.1
	Sample matrices	QAPP Section 4.1

QUALITY ASSURANCE (QA)/R-5 ELEMENTS	COMMENT ADDRESSED IN THIS QAPP
Classification of each measurement parameter as either critical or needed for information only	QAPP Section 5.0
Appropriate validation study information, for nonstandard situations	Not Applicable
B2 Sampling Methods Requirements	QAPP Section 5.0
Identifies sample collection procedures and methods	QAPP Section 5.0
Lists equipment needs	QAPP Section 5.0
Identifies support facilities	QAPP Section 2.0
Identifies individuals responsible for corrective action	QAPP Sections 2.0 and 6.1.5
Describes process for preparation and decontamination of sampling equipment	QAPP Sections 5.1 and 6.1.1
Describes selection and preparation of sample containers and sample volumes	QAPP Section 5.2
Describes preservation methods and maximum holding times	QAPP Section 5.2
B3 Sample Handling and Custody Requirements	QAPP Section 5.3
Notes sample handling requirements	QAPP Section 5.3
Notes chain-of-custody procedures, if required	QAPP Section 5.3
B4 Analytical Methods Requirements	QAPP Section 7.0
Identifies analytical methods to be followed (with all options)	QAPP Section 7.2
Provides validation information for nonstandard methods	Not Applicable
Identifies individuals responsible for corrective action	QAPP Section 6.1.5
Specifies needed laboratory turnaround time	QAPP Section 8.3
B5 Quality Control Requirements	QAPP Section 6.2
Identifies QC procedures and frequency for each sampling, analysis, or measurement technique, as well as associated acceptance criteria and corrective action	QAPP Sections 4.3, 6.2, and 7.0
References procedures used to calculate QC statistics including precision and bias/accuracy	QAPP Sections 4.0 and 6.2
B6 Instrument/Equipment Testing, Inspection and Maintenance Requirements	QAPP Section 6.1.4
Identifies acceptance testing of sampling and measurement systems	QAPP Sections 6.1 and 7.0
Describes equipment preventive and corrective maintenance	QAPP Section 6.1
Notes availability and location of spare parts	QAPP Section 6.1
B7 Instrument/Equipment Calibration and Frequency	QAPP Section 7.0
Identifies equipment needing calibration and frequency for such calibration	QAPP Section 7.0
Notes required calibration standards and/or equipment	QAPP Sections 6.1 and 7.0
Cites calibration records and manner traceable to equipment	QAPP Section 7.0
B8 Inspection/Acceptance of Supplies and Consumables	QAPP Section 6.1
States acceptance criteria for supplies and consumables	QAPP Section 6.1
Notes responsible individuals	QAPP Section 6.1
B9 Data Acquisition For Non-Direct Measurements	Not Applicable
Identifies type of data needed from non-measurement sources (e.g., computer databases and literature files), along with acceptance criteria for their use	Not Applicable
Describes any limitations of such data	Not Applicable
Documents rationale for original collection of data and its relevance to this project	Not Applicable
B10 Data Management	QAPP Section 8.0
Describes standard record-keeping and data storage and retrieval requirements	QAPP Section 8.0
Checklists or standard forms attached to QAPP	QAPP Appendix C

QUALITY ASSURANCE (QA)/R-5 ELEMENTS	COMMENT ADDRESSED IN THIS QAPP
Describes data handling equipment and procedures used to process, compile, and analyze data (e.g., required computer hardware and software)	QAPP Section 8.0
Describes process for assuring that applicable Office of Information Resource Management requirements are satisfied	Not Applicable
C1 Assessments and Response Actions	QAPP Sections 6.1.5 and 8.0
Lists required number, frequency, and type of assessments, with names of responsible personnel (assessments include but are not limited to peer reviews, management systems reviews, technical systems audits, performance evaluations, and audits of data quality)	QAPP Sections 6.1.5 and 8.0
Identifies individuals responsible for corrective actions	QAPP Section 6.1.5
C2 Reports to Management	QAPP Section 8.6
Identifies frequency and distribution of reports for:	
Project status	QAPP Section 8.6
Results of performance evaluations and audits	QAPP Section 8.6
Results of periodic data quality assessments	QAPP Section 8.6
Any significant QA problems	QAPP Section 8.6
Preparers and recipients of reports	QAPP Section 8.6
D1 Data Review, Verification, and Validation	QAPP Section 8.4 and Appendix C
States criteria for accepting, rejecting, or qualifying data	QAPP Section 8.4 and Appendix C
Includes project-specific calculations or algorithms	Not Applicable
D2 Verification and Validation Methods	QAPP Section 8.4 and Appendix C
Describes process for data validation and verification	QAPP Section 8.4 and Appendix C
Identifies issue resolution procedure and responsible individuals	QAPP Section 8.4
Identifies method for conveying these results to data users	QAPP Section 8.4
D3 Reconciliation with User Requirements	QAPP Section 8.4
Describes process for reconciling project results with Data Quality Objectives (DQOs) and reporting limitations on use of data	QAPP Section 8.4

## QUALITY ASSURANCE PROJECT PLAN

URS Corporation (URS) has prepared this Quality Assurance Project Plan (QAPP) on behalf of the Arizona Department of Environmental Quality (ADEQ) for the Miracle Mile Water Quality Assurance Revolving Fund (WQARF) Site in Tucson, Arizona (hereafter referred to as the Site). The purpose of this QAPP is to designate and document the specifications and methods that will be employed to establish technical accuracy and precision, statistical validity, and documentary evidence of data generated during a soil sampling event conducted at the Site.

URS has prepared this QAPP to address quality assurance/quality control (QA/QC) policies and procedures associated with the collection of environmental data at the Site. The QAPP provides field and laboratory personnel with instructions regarding activities to be performed before, during, and after field sampling activities. These instructions are intended to ensure data collected for use in project decisions will be of the type and quality needed and expected for their intended purpose.

Guidelines followed in the preparation of this QAPP are described in the following Environmental Protection Agency (EPA) and ADEQ documents: EPA Requirements for Quality Assurance Plans, EPA QA/R-5 (EPA, 2001); EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5 (EPA, 2002); Guidance for the Data Quality Objectives Process, EPA QA/G-4 (EPA, 2006); and ADEQ Superfund Program Section Quality Assurance Program Plan (ADEQ, 2000).

The QAPP will be approved and distributed to ADEQ, URS, and Accutest Laboratories (Accutest) staff listed on the signature page (Page i). Addenda and/or revisions to this QAPP can be initiated by all parties listed on the signature page; however, the appropriateness of an addendum or revision is determined by the URS project manager.

## 1.0 PROJECT OVERVIEW

Although the sampling program is formally described in the Work Plan (URS, 2012), this section includes a brief overview of the project and its history.

The Site is located in northwest Tucson, Arizona and was originally was part of the Miracle Mile Interchange Area (MMIA). The Site was placed on the WQARF priority list on November 13, 1987, based on the detection of chlorinated volatile organic compounds (VOCs) in water supply wells located within the boundaries of the MMIA. The MMIA extended from Wetmore Road on the north to Speedway Boulevard on the south, and from Oracle Road on the east to Silverbell Road on the west. Based on the findings from subsequent investigations, ADEQ listed a smaller area on the WQARF Registry in 1988 and this area became known as the “Miracle Mile WQARF Site.” Continued investigation, including the Remedial Investigation (RI), has resulted in the boundaries of the Site being reduced to a smaller area and defined by releases that have occurred in the vicinity of Romero Road, between Prince Road and Roger Road.

In 2003, Spring Joint Specialists, Inc. hired a contractor to remove the underground storage tanks (USTs), piping, concrete slabs, and contaminated soils associated with the sump and chrome tanks located on the property. Contaminated soil with hexavalent chromium levels as high as 3,420 milligram per kilogram (mg/kg) were excavated from the site and disposed of as hazardous waste.

## 2.0 PROJECT/TASK ORGANIZATION

This section identifies the individuals and organizations participating in the project and discusses their specific roles and responsibilities. The participants discussed include, but may not be limited to, the principal data users, decision makers, project QA officers, and persons responsible for the implementation of the plan.

***ADEQ Project Manager (Ms. Gretchen Wagenseller)*** – Ms. Wagenseller maintains overall responsibility for the direction of the scope of work to be performed for the project. She provides final review and approval of documents, reports, plans, schedules, and other communications submitted pursuant to a task assignment. Further, Ms. Wagenseller provides overall coordination of the project and provides the consultant with an overview and with direction.

***URS Project Manager (Mr. William Neese)*** – Mr. Neese is responsible for the management of the project, which includes scheduling, staffing, budget compliance, and field operations. In addition, Mr. Neese provides senior technical review of project documents. He directs and supervises task and field managers, and confirms that all personnel understand the scope of work and QA/QC requirements. Mr. Neese is also responsible for client satisfaction with the services provided.

***URS QA Officer (Ms. Marianne Burrus)*** – Ms. Burrus has the responsibility to ensure all laboratory procedures follow those protocols established in the QAPP and meet the regulatory guidance. Ms. Burrus' additional responsibilities for the project include coordinating data receipt from the laboratory and performing data verification/validation tasks. If the URS QA officer determines that laboratory procedures do not adhere to the established protocols and the data integrity may be impacted, it is her responsibility to inform the URS project manager.

***Accutest, QA Manager (Ms. Guergana Gueorguieva)*** – Ms. Gueorguieva has the responsibility to ensure that the laboratory protocols are being followed as required by the QAPP. The Accutest QA manager (or her designee) shall review each chemical data package before submission to URS. It is the responsibility of the Accutest QA manager to report any technical deficiencies to the Accutest laboratory manager and to implement corrective actions. Ms. Gueorguieva is responsible for informing the Accutest project manager of any issues that may impact data quality. This includes deviations from established protocols.

### 3.0 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATION

The field activities included as part of this investigation consists of soil sampling. Personnel completing these activities have sufficient training to follow the procedures required for the activities listed in the Work Plan. These activities are considered standard and do not require specialized training. All field staff have Hazardous Waste Operations Emergency Response training as described in 29 Code of Federal Regulations 1910.120. Documentation of the training is filed with the URS health and safety officer.

The analytical laboratory for this project is Accutest located in Phoenix, Arizona. Accutest is licensed by the Arizona Department of Health Services (ADHS) “Office of Laboratory Licensure, Certification, and Training” (as described in A.A.C. R9-14-601 through R9-14-618) for all the analytical methods required for this sampling event. Accutest performs under the licensure number AZ0762.

## 4.0 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) have been specified for each data collection activity, and the work will be conducted and documented so that the data collected are of sufficient quality for their intended use (EPA, 2002). DQOs specify the data type, quality, quantity, and uses needed to make decisions, and are the basis for designing data collection activities. The DQOs have been used to design the data collection activities presented in the Work Plan (URS, 2012). The DQOs for the project are discussed in the following sections.

### 4.1 DATA QUALITY OBJECTIVE PROCESS

The project DQOs developed specifically for the planned sampling and analysis program have been determined based on the EPA's seven-step DQO process (EPA, 2006). The project manager will evaluate the project DQOs to determine if the quantitative and qualitative needs of the sampling and analysis program have been met. The project definition associated with each step of the DQO process can be summarized as follows:

#### 1. State the Problem:

Historical releases from a chromium plating facility in the vicinity of the Miracle Mile WQARF site have released hexavalent chromium into the soil with potential to move to the groundwater.

#### 2. Identify the Goal of the Study:

The goals are to quantify the extent of hexavalent chromium in the soil and in the groundwater and propose possible future treatment remedy options for chromium in groundwater.

#### 3. Identify Information Inputs:

Soil core samples will be used to evaluate the current chromium distribution.

#### 4. Define the Boundaries of the Study:

The study area is defined as described in Section 1.0 and can also be found in the Spring Joint Specialists chromium contaminated soil characterization work plan figures. The nine boreholes will be located at or near the former Spring Joint Specialists property located at 3660 North Romero Road in Tucson, Arizona. The site is located within the boundaries of the Miracle Mile WQARF area.

## **5. Develop the Analytic Approach:**

The concentration of hexavalent chromium will be compared to soil remediation levels. Depth-specific sampling in selected boreholes will be used to evaluate potential impacts to perched groundwater.

## **6. Specify Performance or Acceptance Criteria:**

The results of all analytical testing will be subjected to data verification. Data are determined to be valid if the specified limits on precision, accuracy, completeness, representativeness, comparability, and sensitivity are achieved. Outlier field or laboratory results may be evaluated using an appropriate statistical test. The quality of the data will be assessed through the procedures further described in this QAPP.

## **7. Develop the Plan for Obtaining Data:**

There will be nine boreholes drilled to 99 feet each, or until perched groundwater is reached. Samples will be collected every 5 feet from the continuous cores produced from the rotosonic drilling for a total of approximately 180 samples.

## **4.2 DATA CATEGORIES**

The two general categories of data are defined as (1) definitive data and (2) screening data. Definitive data are generated using rigorous analytical methods, such as approved EPA or ASTM reference methods. Definitive data are analyte specific, and both identification and quantification are confirmed. These methods have standardized QC and documentation requirements and the data are not restricted in their use unless quality problems require data qualification.

The definitive data will be generated by Accutest located in Phoenix, Arizona. Accutest is licensed by ADHS “Office of Laboratory Licensure, Certification, and Training” (as described in A.A.C. R9-14-601 through R9-14-618) for all the analytical methods required for this sampling event.

## **4.3 DATA QUALITY INDICATORS**

This section identifies the data quality indicators for the DQOs; defines the elements of the QC program for field operations and laboratory analyses; and defines the requirements for precision, accuracy, completeness, representativeness, comparability, and sensitivity.

### 4.3.1 Precision

Precision measures the reproducibility of repetitive measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the sample process under similar conditions.

Precision is evaluated by measuring the agreement among individual measurements of the same property under similar conditions. Generally, field precision is measured through the collection and analysis of field duplicate samples and laboratory precision is measured through the analysis of duplicate control samples (LCSD) and matrix spike duplicates (MSD). Total precision is a measurement of the variability associated with the entire sampling and analytical process.

Duplicate results are assessed using the relative percent difference (RPD) between duplicate measurements. The RPD will be calculated as follows:

$$RPD = (200)(X_1 - X_2) / (X_1 + X_2)$$

where,  $X_1$  is the larger of the two observed values, and  $X_2$  is the smaller of the two observed values.

Laboratory precision will be measured through the analysis of LCSD and MSD. Evaluation of the RPD will be based on the criteria given in the applicable analytical method. If no established limit is available, the RPD limit of 30 percent will be applied. Data not meeting the control limit will be qualified as described in the applicable data review procedure.

### 4.3.2 Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systematic error. It reflects the total error associated with a measurement. Simply put, accuracy is the measure of closeness of data to their true values. A

$$\%R = (100)(X_s - X) / T$$

measurement is accurate when the value reported does not differ beyond acceptable limits from the true value or known concentration of the spike or standard. Laboratory accuracy is expressed as the percent recovery (%R), which is calculated as follows:

Field accuracy will be assessed through the collection and analysis of field equipment blanks. Field equipment blanks (rinsates) are not required when dedicated sampling equipment is employed. However, in the event non-dedicated equipment is used rinsates will be collected at a frequency of 10 percent.

Analytical accuracy will be assessed by initial and continuing calibration of instruments and the analysis of laboratory control samples (LCS) and matrix spikes (MS), where applicable. Evaluation of the RPD will be based on the criteria given in the analytical methods. Data not meeting the control limit will be qualified as described in the applicable data review procedure.

### 4.3.3 Completeness

Completeness is the amount of valid data obtained compared to the amount that was expected under ideal conditions. The number of valid results divided by the number of possible results, expressed as a percentage, determines the completeness of the data set. The formula for calculation of completeness is, as follows:

$$\% \text{ completeness} = (100) \times \left( \frac{\text{number of valid results}}{\text{number of possible results}} \right)$$

Field completeness is a measure of the amount of valid field measurements obtained from all field measurements taken during the sampling events. The completeness goal for field measurements and sample collection is 100 percent. If field measurements are not collected from a specific location or a sample was not collected due to environmental conditions, the completeness for field activities will be identified in the field notes.

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements completed at the laboratory. Data qualified by the laboratory or data reviewer as estimated are considered usable and, therefore, complete. The completeness goal for laboratory activities, is 95 percent. The requirement for holding times will be 100 percent.

### 4.3.4 Representativeness

Representativeness is the degree to which data accurately and precisely represent selected characteristics of the media sampled. Representativeness of data collection is addressed by preparation of sampling and analysis programs.

This QAPP, together with the Work Plan, addresses representativeness of field data by specifying sufficient and proper numbers and locations of samples incorporating appropriate sampling methodologies specifying proper sample collection techniques and decontamination procedures and establishing proper field QA/QC procedures.

Representativeness in the laboratory is ensured by using proper analytical procedures and appropriate methods, meeting analytical holding times, and meeting QC criteria for each method. It is the laboratory project and QA manager's responsibility to ensure that the proper methods and criteria are employed by the laboratory. Any deviation from the QAPP or laboratory Standard Operating Procedures (SOP) will be noted in the case narrative.

#### **4.3.5 Comparability**

Comparability is an expression of confidence with which one data set can be compared to another. The objective of comparability is to ensure that data developed during the investigation are comparable to site knowledge and adequately address applicable criteria or standards established by EPA and ADEQ.

The comparability goal is achieved through the use of standard field techniques. These include, but are not limited to, the project prescribed techniques for sample collection and field parameter measurements. A detailed description of field techniques is given in the Work Plan. It is the URS field manager's responsibility to ensure that the proper field techniques and procedures are adhered to. Any deviation from the Work Plan will be noted in the field notes.

The comparability of laboratory data will be ensured by the laboratory personnel having reviewed the QAPP and having a working knowledge of the analytical SOP. The laboratory QA manager (or designee) will also ensure comparable data by reviewing all data generated, and verifying that the correct methods have been used. The data reviewer will also review the data to ensure compliance with the various method requirements.

#### **4.3.6 Sensitivity**

Sensitivity is a measure of the analytical detection or quantification limits. A detection is the minimum amount of analyte that can be consistently measured and reported with a high degree of confidence that the analyte concentration is above background response. A quantification limit is that amount that can be consistently quantified with acceptable precision and accuracy. This is also referred to as a practical quantitation limit.

The laboratory practical quantitation limit will be established and verified as outlined in the analytical methods and in accordance with ADHS laboratory licensure rules. Where applicable, the laboratory quantitation limits required for this project must be below residential Soil Remediation Levels (res-SRLs).

## 5.0 SAMPLE PROCESS DESIGN

The defensibility of data is dependent on the use of well defined, accepted sampling procedures. This section describes the sampling and handling procedures that will be followed for each sampling event.

### 5.1 FIELD PROCEDURES

Collection of environmental samples of high integrity is important to the quality of chemical data to be generated. To this end, strict field procedures have been developed as general descriptions of field methods that will be employed at various locations during phases of the field investigation. All soil samples will be collected according to the procedures outlined in the Work Plan. The number of soil samples to be collected, their locations, and sample design and rationale are also included in the Work Plan.

### 5.2 FIELD ANALYSIS

The only analysis that will be conducted in the field is VOC analysis in soil cores with the photoionization detector. If there are any positives the soil will be sent to the lab for verification.

### 5.3 SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

Each environmental compound has specifications about the containers the sample is kept in, the preservation method and what the holding time a sample is allowed before it must be processed. This is to ensure stability of the compound as well as for safety of the sampler.

The sample container size and quantity hexavalent chromium as indicated by the EPA is a minimum of 10 grams of soil collected in a glass or plastic container, there can be no metal in the sample container. If the container is glass it should have a Teflon coated lid, however plastic is recommended because metals can sometimes sorb to glass surfaces. Required preservation for hexavalent chromium soil samples are at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  for a maximum of 30 days. The laboratory will provide to URS new sample jars as needed for sample collection. Sample containers provided by the laboratory will be purchased commercially from I-Chem, Eagle Pitcher, or equivalent source. In addition, the laboratory will maintain the “certificate of cleanliness” for the containers should any questions arise in the future.

## 5.4 SAMPLE CUSTODY

Custody of samples will be maintained and documented from the time of sample collection to completion of the analyses. A sample is considered to be under a person's custody if one or more of the following conditions are met:

- the sample is in the person's physical possession
- the sample is in view of the person after that person has taken possession
- the sample is secured by that person so that no one can tamper with the sample
- the sample is secured by that person in an area that is restricted to authorized personnel

### 5.4.1 Field Custody Procedures

Upon collection, samples will be considered to be in the sampler's custody. The sampler will be responsible for the care and custody of the samples until they are relinquished for delivery to the laboratory or accepted by the laboratory. Samples will be collected and named according to the procedures described in the Work Plan.

The analytical laboratory will provide chain-of-custody (COC) forms, cooler custody seals, and sample labels, which will be completed by the field technician. The sample label will include the job number, unique sample identification (ID), date and time of sample collection, type of analysis requested, and type of preservative used in the sample container(s).

Samples will be accompanied to the laboratory by a COC form, which will contain the following information:

<b>PROJECT NAME</b>	<b>SAMPLE NUMBERS</b>
<b>DATE OF COLLECTION OF SAMPLES</b>	<b>TIME OF COLLECTION OF SAMPLES</b>
<b>SAMPLE MATRIX DESCRIPTION</b>	<b>ANALYSES REQUESTED FOR EACH SAMPLE</b>
<b>PRESERVATION METHOD, IF APPLICABLE</b>	<b>NUMBER AND TYPE OF CONTAINERS USED</b>
<b>ANY SPECIAL HANDLING OR ANALYSIS REQUIREMENTS</b>	<b>SIGNATURE OF PERSON COLLECTING THE SAMPLES</b>

**SIGNATURE(S) OF PERSONS INVOLVED IN  
THE SAMPLE CUSTODY.**

The COC form will be filled out in indelible ink. When the samples are transferred from one party to another, the individuals will sign, date, and note the time on the form. The original form will accompany the sample delivery to the laboratory in the shipping cooler. The sampling personnel will retain a copy of the form. A copy of the Laboratory's COC is included in Appendix A.

The following procedures will be used (as applicable) when packing and transporting samples to the laboratory:

- use of waterproof ice chests and coolers
- use of blue ice and frozen water (e.g., cubes, shaved) to maintain proper refrigeration of the samples ( $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ )
- cushioning material placed within the cooler
- paperwork placed inside a waterproof bag inside the cooler
- the cooler lid taped closed with packaging tape

Samples will be transported as soon as possible after sample collection to the laboratory for analysis. The field manager will notify the laboratory in advance of sample delivery. Sample coolers will be delivered via commercial carrier to the laboratory. A copy of the COC will be retained by URS.

#### **5.4.2 Laboratory Custody Procedures**

Upon arrival at the laboratory, the laboratory will check samples for label identifications and complete, accurate COC documentation. The sample condition will be checked and the temperature will be measured immediately after the cooler is opened. Any discrepancies between the COC documentation and sample labels, inaccurate or incomplete sample preservation, or any problem encountered that may affect the sample integrity must be noted and communicated to the URS project manager or QA officer.

A unique laboratory ID number will be assigned. This number will be cross-referenced to the field sample ID in an attempt to deter the possibility of mislabeling. Analytical reports will contain both ID numbers for samples results. Access to the sample control area will be restricted to prevent any unauthorized contact with samples, extracts, or documentation.

All samples and sample extracts will be maintained by the laboratory until 30 days following the release of the final report.

## 6.0 QUALITY CONTROL ELEMENTS

This section presents QC requirements relevant to the collection and analysis of environmental samples that will be followed during all project analytical activities. The purpose of the QC program is to produce data of known quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials.

### 6.1 QUALITY CONTROL PROCEDURES

The chemical data to be collected for this effort will be used to determine the vertical extent of cadmium, lead and total petroleum hydrocarbon contamination. In addition, these data will also be used to obtain a no further action determination; therefore, data of the highest confidence and quality are critical. Consequently, strict QA/QC procedures will be adhered to. These procedures include:

- Adherence to strict protocols for field sampling and decontamination procedures;
- If required, collection and laboratory analysis of appropriate field equipment blanks to monitor for cross-contamination of samples in the field;
- Collection and laboratory analysis of matrix spike, matrix spike duplicate, and field duplicate samples to evaluate precision and accuracy; and
- Attainment of completeness goals.

#### 6.1.1 Equipment Decontamination

Sampling equipment, including trowels and hollow stem augers, will be decontaminated using clean water containing a laboratory-grade detergent, followed by a double rinse with distilled water before each sample collection. Decontaminated equipment will be allowed to air dry, or will be dried with lint free towels.

#### 6.1.2 Standards

Standards used for calibration or to prepare samples will be certified by the National Institute of Standards and Testing, EPA, or other equivalent source. The standards will be current. The

expiration date will be established by the manufacturer, or based on chemical stability, the possibility of contamination, and environmental and storage conditions. Standards will be labeled with expiration dates, and will reference primary standard sources if applicable. Expired standards will be discarded. It is the laboratory's responsibility to ensure that all standards used meet the requirements outlined above.

### **6.1.3 Supplies**

All supplies will be inspected prior to their use in the field or laboratory. The descriptions for sample collection and analysis contained in the methods will be used as a guideline for establishing the acceptance criteria for supplies. A current inventory and appropriate storage system for these materials will ensure their integrity prior to use. Efficiency and purity of supplies will be monitored through the use of standards and blank samples. For laboratory supplies it is the laboratory's responsibility to ensure that all supplies meet the requirements outlined above. For field supplies, it is the URS field manager's responsibility to ensure that all supplies meet the requirements outlined above.

### **6.1.4 Preventive Maintenance**

Most field instruments operate by solid-state circuitry and require little preventive maintenance, other than cleaning. However, general inspection and maintenance will be conducted in accordance with manufacture's recommendations. Preventive maintenance will be documented in each instrument's field notebook by the equipment manager or the person conducting the maintenance. URS will maintain a stock of spare parts and consumables for field analytical equipment.

Preventative maintenance will be performed for each analytical instrument to minimize improper performance or interruption of the analytical process. General inspection and maintenance will be conducted in accordance with manufacture's recommendations. Designated laboratory personnel or outside-service contracted firms will be responsible for this maintenance. The laboratory will maintain a stock of spare parts and consumables for analytical equipment. All routine maintenance and specialized repairs will be documented in a bound maintenance notebook with sequentially numbered pages. Each analytical instrument will have its own maintenance notebook. Entries will be initialed, dated, and periodically reviewed by appropriate personnel. Accutest's preventative maintenance procedures are documented in their Quality Systems Manual (QSM).

### 6.1.5 Corrective Actions

The URS project manager is responsible for the initiation and implementation of corrective actions with respect to the field sampling operations and it is his responsibility to see that field sampling procedures are followed.

Corrective actions may include the following:

- training field personnel
- modifying field procedures
- re-sampling project sample locations

The laboratory's QA officer, in consultation with the URS QA officer, is responsible for implementing corrective actions in the laboratory (from sample receipt to final data deliverable). It is their combined responsibility to see that analytical and sampling procedures are followed as specified and that the data generated meet the acceptance criteria.

Corrective actions for the laboratory may include the following:

- reanalyzing and/or re-extracting samples
- correcting laboratory procedures
- recalibrating instruments using freshly prepared standards
- replacing solvents or other reagents that give unacceptable blank values
- training laboratory personnel in correct sample preparation and analysis procedures

Whenever corrective action is deemed necessary, the analytical laboratory will check that the following steps are taken:

- the problem is defined
- the cause of the problem is investigated and determined
- appropriate corrective action is determined
- corrective action is implemented and its effectiveness verified
- control is reestablished to the noncompliant QC parameter(s)

The corrective actions will be documented according to the LAB QSM, (Appendix A).

## **6.2 QUALITY ASSURANCE AND QUALITY CONTROL SAMPLES**

The purpose of this QA/QC program is to produce data of known quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials. QC samples will be collected as part of the overall QA/QC program.

The data quality indicators for the measurement of field data including the field objectives for precision, accuracy, completeness, representativeness, comparability, and sensitivity are outlined in Section 4.3 and briefly listed below.

The laboratory QC samples have been selected based on the DQOs for this project and the established analytical method requirements. The required laboratory QC samples are outlined below; however, additional QC samples may be required by the laboratory in order to satisfy the internal QC policies. The acceptance criteria for the laboratory QC samples are given in Appendix A.

### **6.2.1 Equipment Blank**

The equipment blanks that we will be completing in the field are for the soil sampling equipment. We will be using corers, trowels and bowls. These should all be tested in the field to check for possible contamination.

### **6.2.2 Method Blank**

A method blank is a sample of ASTM Type II or analyte-free (deionized) water that is carried through each step of the preparation and analytical method. A method blank sample is required for each analytical batch of 20 or fewer samples. Method blank samples are used to assess potential contamination attributed to laboratory operations during sample preparation and analysis.

### **6.2.3 Instrument Blank**

An instrument blank is a sample of ASTM Type H or analyte-free (deionized) water that is analyzed with associated calibrations of laboratory instruments. Instrument blank results are used to assess potential contamination attributed to specific instrument calibration procedures.

#### **6.2.4 Surrogate Spikes**

Surrogate spikes (also known as System Monitoring Compounds) are compounds added to every blank, standard, sample, and matrix spike sample as specified in the analytical methodology. Surrogate compounds are generally brominated, fluorinated, or isotopically labeled compounds not expected to be present in environmental samples. The results of the surrogate spike compounds are used to evaluate the accuracy of the analytical measurement on a sample-specific basis. Surrogate spikes are generally added for organic analyses only.

#### **6.2.5 Internal Standards**

An internal standard is a standard of known concentration added to each sample and carried through the entire determination procedure as a reference for calibrating and controlling the precision bias of the analytical method. Internal standards are generally used for organic analyses only.

#### **6.2.6 Matrix Spikes and Matrix Spike Duplicates**

MS are known concentrations of analytes added to a sample and carried through each step of the preparation and analytical method. MS are typically analyzed in duplicate (MSD) for organic analyses. The results of MS are reported in %R and are evaluated to assess potential matrix interferences. The results of MSD are reported as RPD and are evaluated to assess laboratory and method precision.

#### **6.2.7 Matrix Duplicates**

A matrix duplicate (or laboratory duplicate) is a separate aliquot of a sample taken from the sample container and carried through each step of the preparation and analytical method. The results of matrix duplicates are reported as RPD and are evaluated to assess laboratory and method precision.

#### **6.2.8 Laboratory Control Samples/Laboratory Control Sample Duplicate**

LCS and LCSD are well-characterized, laboratory-generated samples used to monitor the laboratory's day-to-day performance of analytical methods. The LCS may be a purchased standard, or a method blank spiked with known concentrations of target analytes. The LCS is carried through each step of the preparation and analytical method. LCS should be reported in %R and used to assess the accuracy and precision (use of LCSD) of the analytical process

independent of matrix effects. Controlling lab operations with LCS (rather than surrogates or MS) offers the advantage of being able to differentiate low recoveries due to procedural errors with those due to matrix effects.

### **6.2.9 Continuing Calibration Verification**

Continuing calibration verification (CCV) is achieved by the routine analysis of a standard of known concentration. The verification standard concentration is usually at or near the midpoint of the linear calibration curve. CCV for linear calibrations involves the calculation of the percent drift or percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The ADEQ policy for the use of single point calibrations and continuing calibration verification constraints is included in Appendix B.

## **7.0 ANALYTICAL AND CALIBRATION PROCEDURES**

This section identifies the analytical methods used during the analysis of samples and calibration procedures and frequencies for field and laboratory instruments that will be used for the collection of data.

### **7.1 FIELD ANALYTICAL METHODS AND CALIBRATION**

When quantitating samples, the technician will ensure that the appropriate QC is instituted at the appropriate times with the appropriate frequency. At a minimum, maintenance intervals for field instruments will be those recommended by the respective manufacturers, unless experience dictates a shorter interval. Calibrations for field instruments will be performed at the beginning and end of each day and recorded in a field notebook. Adherence to the calibration schedule is mandatory. The fact that these calibrations may be performed by an outside source does not exempt the user from the responsibility for identifying, monitoring, and controlling calibration intervals and ensuring that maintenance checks are made on time.

### **7.2 LABORATORY ANALYTICAL METHODS AND CALIBRATION**

The EPA methods used for analysis of hexavalent chromium are Methods 3060A, 7199 and 7196A. Method 3060A is used for the alkaline digestion for hexavalent chromium and is needed to extract the chromium from the soil into a liquid for analysis. The analysis is done colorimetrically by Method 7199 using Ion Chromatography or Method 7196A using UV-VIS Spectrophotometry. The general laboratory QA procedures are provided in the Accutest Quality Systems Manual (Appendix A). The SOPs for Method 7196A are also included in Appendix A.

Instrumentation and equipment used during sample analysis will be operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations, as well as criteria set forth in the applicable analytical methodology references. Operation, calibration, and maintenance will be performed by personnel properly trained in these procedures. Laboratory capabilities will be demonstrated initially for instrument and reagent/standards performance as well as accuracy and precision of analytical methodology.

Calibration will be required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet required reporting limits. Instruments will be calibrated with standard solutions appropriate to the type of instrument and the linear range required for the DQOs of this project. The frequency of initial calibration and calibration

verification will meet the requirements of the analytical method. Calibration procedures for instruments and the acceptance criteria are summarized in the analytical methods. Samples must be bracketed by passing calibration check standards where required by the method.

Calibration standards and acceptance criteria vary depending on the instrument and analytical method. The general principles of calibration will apply uniformly. Initial calibration will demonstrate the reporting limits, the dynamic range of the detection system, and the retention windows. EPA procedures outline each system's acceptance criteria for calibration prior to analyses. Initial calibration consists of the analysis of at least five calibration standards at varying concentrations. The low calibration standard will be at a concentration below or at the reporting limit. The other standards will be at concentrations in the expected range of the detection system. The results will be used to determine a calibration curve and response factors for each analyte. The sample concentration (diluted or undiluted) will not exceed the linear range determined by the initial calibration.

CCV standards are analyzed before analysis of field samples and after every 10 samples to verify the initial calibration curve and response factors or at a frequency as required in the method. Initial calibration and CCV standards will contain all analytes of interest and must meet all calibration criteria. The ending or bracketing calibration standards also must meet the criteria where required by the method.

## **8.0 DOCUMENT AND DATA MANAGEMENT**

This section identifies the process and responsibilities for ensuring that the appropriate project personnel have the most current approved version of the QAPP and other project documents noted, including revisions and addenda. In addition, this section presents reporting requirements and data handling procedures relevant to the data produced during all project analytical activities.

### **8.1 QAPP DISTRIBUTION**

The URS project manager and QA officer are responsible for ensuring that each project member has access to the most current version of the project QAPP, including all subsequent addenda or revisions. The project members include, but may not be limited to, all parties indicated on the signature page of this report.

### **8.2 RECORDS DISPOSITION**

All project files and records will be stored on site at URS until the no further action determination has been granted by ADEQ. The project files will then be moved to an off-site storage facility for permanent storage. Project information can be obtained through a written request to the URS project manager. The requested information should be made available within seven working days.

Accutest will store the original hardcopy and electronic raw data of the analytical data packages produced for this project for five years. The level of information regarding sample analyses (calibration records, run logs, etc.) will be such that the analytical processes can be reconstructed within that time frame.

### **8.3 DATA REPORTING**

URS office data management will involve establishing and maintaining a project file. The project file will be maintained by the document control personnel. Project-related information (including field and laboratory documents and data) will first be routed to the URS project manager, who then will be responsible for routing the information to appropriate personnel.

Field observations and measurements will be recorded in field notebooks. The field data will be reviewed by the project manager to evaluate completeness of the field records and appropriateness of the field methods employed. Documentation in the field logbook will be sufficient to reconstruct the sampling situation without relying on the memories of the field team

members. A detailed description of all field data entries required for this sampling event is given in the Work Plan

Accutest will provide the analytical reports within 15 working days of sample receipt. Analytical data will contain the necessary sample results and quality control data to evaluate the DQOs defined for this project. Omissions or insufficient levels of detail will be corrected at the expense of the laboratory. Documentation requirements for laboratory data should include, at a minimum, the following data and summary forms:

- narrative (including a complete description of any difficulties or QA/QC deficiencies encountered during sample analysis), cross reference, COC, and method references
- analytical results with cross reference to analytical batch
- surrogate recoveries (as applicable)
- blank results
- Laboratory Control Sample recoveries
- sample spike recoveries
- duplicate sample results or duplicate spike recoveries

In addition, the laboratory must qualify all outliers according to ADEQ Data Qualifiers (see Appendix B). The following sections outline the requirements for their submission and the handling of these deliverables.

#### **8.4 PROCEDURES FOR DATA REVIEW**

Data validation refers to a review of 100 percent of the data generated (including raw data). This level of review requires that data documentation consistent with EPA Level IV be received from the laboratory. This level of review will not be performed unless requested by ADEQ or if determined by URS, laboratory/data quality issues arise that may affect the ultimate use of the data. If laboratory/data quality issues do arise and an EPA Level IV package is deemed necessary, 100 percent of the affected data will undergo data validation.

Data verification refers to all other levels of review (other than validation). It is anticipated that 99 percent of the analytical data collected for this project will undergo the data verification

procedure outlined below. All data collected for this project will undergo data verification, at a minimum.

Guidance for performing data verification for the types of analyses to be utilized for this investigation is provided in the *National Functional Guidelines* (EPA, 2004). Data verification will be documented in a manner consistent with these functional guidelines. It is the URS QA Officer's responsibility to verify 100 percent of the data as described below and to determine if data validation (as described above) is necessary.

The laboratory data will be reviewed for compliance with meeting the QA/QC specifications outlined in the analytical methods and this QAPP. The following summarizes the areas of data verification, where applicable:

- data completeness including COC documentation
- holding times and sample preservation
- blanks
- surrogate recoveries
- LCS
- Matrix Spike / Matrix Spike Duplicate (MS/MSD)
- laboratory duplicates
- field duplicate samples
- other QA/QC issues documented in the data deliverable

The application of data verification criteria is a function of project-specific DQOs. The URS QA officer will determine if the DQOs for the analytical data have been met, review the data according to the URS verification procedures (Appendix C), and apply applicable data validation flags, if any.

## **8.5 DATA QUALIFIERS**

Data verification/validation flags are applied to those sample results that fall outside of the acceptance criteria specified in the analytical methods, laboratory SOP, and this QAPP and, therefore, did not meet the program's quality goals. Data validation flags to be used for this

project are defined in the National Functional Guidelines. Data validation flags will indicate if results are considered anomalous, estimated, or rejected. Only rejected data are considered unusable for decision-making purposes; however, other qualified data may require further verification.

Results of the data verification/validation review will be documented and summarized in a Data Verification/Validation Memorandum. The memorandum is submitted to the URS project manager for his review. It is the URS project manager's responsibility to communicate the results of the data verification/validation process to the ADEQ project manager.

## **8.6 REPORTS TO MANAGEMENT**

The following section describes the reports issued during these sampling events to inform management of the status of the project and results of the analytical data.

- The completed field forms must be submitted to the URS field manager at the end of each sampling day.
- The laboratory data deliverables must be submitted to the URS project manager within 10 working days after sample receipt.
- The URS QA officer will submit QA reports (for example, laboratory audits and/or results for performance evaluation samples) to the URS project manager in a timely manner.

The URS project manager will submit a report summarizing the results of the investigation to the ADEQ project manager within approximately 45 days after laboratory results have been received. The report will summarize field activities, observations, and problems encountered, and may provide recommendations for future sampling rounds. This report is to include the following:

- field notes and forms (including data sheets)
- laboratory reports
- Data Verification/Validation Memorandum
- tabulated results of laboratory analyses (anomalous results and conditions will be addressed, if required).

## 9.0 REFERENCES

- Arizona Department of Environmental Quality (ADEQ). May 22, 2000. Superfund Program Section Quality Assurance Program Plan.
- U.S. Environmental Protection Agency (EPA). 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA QA/G-4. Office of Research and Development U.S. Environmental Protection Agency. Washington, D.C.
- \_\_\_\_\_. 2002. EPA Guidance for Quality Assurance Project Plans. EPA/240/R-02/009 (EPA QA/G-5). Office of Research and Development U.S. Environmental Protection Agency. Washington, D.C.
- \_\_\_\_\_. 2001. EPA Requirements for Quality Assurance Project Plans. EPA/240/B-1/003 (EPA QA/R-5). Washington, D.C.
- \_\_\_\_\_. 2004. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. EPA540/-R-04-004. Superfund Remediation and Technology Innovation. Washington, D.C.
- URS Corporation. 2002. Work Plan, Shallow Soil Removal Action. 4484 E. Tennessee Street, Tucson, Arizona.

**APPENDIX A**  
**ACCUTEST QUALITY SYSTEMS MANUAL**  
**STANDARD OPERATING PROCEDURE – METHOD 7196A**  
**CHAIN OF CUSTODY**

SOP No: GEN009-3  
 Effective Date: 10/05/2012  
 Replaces: GEN009-2\_03/22/2010  
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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**
**Approvals:**

	See the QA Dept Hardcopy for Signature		
Approved by:	Kesavalu Bagawandoss – Laboratory Director	Date:	
	See the QA Dept Hardcopy for Signature		
Approved by:	Guergana Gueorguieva – QA Manager	Date:	

**Annual Review**

Reviewed by:	Pedro Hufano	Date:	10/01/2012
Reviewed by:		Date:	
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Reviewed by:		Date:	

**Document Control**

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Gen Chem Dept	10/05/2012	-01
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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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**Title: Hexavalent Chromium in Soils by Alkaline Digestion SW 3060A  
Followed by Method 7196A Colorimetric**

Reference: SW846 Method 3060A, Revision 1, Update III, December 1996 Alkaline Digestion  
SW846 Method 7196A, Revision 1, July 1992, Colorimetric

Revision(3): Section 2.3; 7.16.1;7.16.2;9.2.2;9.2.5;10.2

### **1.1 SCOPE AND APPLICATION**

- 1.1 This method is used to determine the concentration of hexavalent chromium in soils, sludges, brick, concrete, and other solid matrices. The solid sample is digested in an alkaline digestion solution to solubilize both water soluble and water insoluble hexavalent chromium compounds. Magnesium chloride in a phosphate buffer is added to suppress oxidation of Cr(III).The hexavalent chromium is determined in the digestate by reaction with diphenylcarbazide in acid solution. The diphenylcarbazide complex produces a characteristic pink color which can be measured spectrophotometrically at 540 nm.
- 1.2 The method reporting limit is 1.0 mg/Kg in solid.

### **2.0 SUMMARY OF METHOD**

- 2.1 The method uses an alkaline digestion to solubilize both water-insoluble and water soluble Cr (VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during the digestion procedure. Failure to meet the pH specification will need for redigestion of the samples.
- 2.2 The sample is digested using 0.28M Na<sub>2</sub>CO<sub>3</sub> / 0.5M NaOH solution and heating a 90-95°C for 60 minutes to dissolve the Cr (VI) and stabilize it against reduction to Cr (III).
- 2.3 The Cr (VI) reaction with diphenylcarbazide is the most common and reliable method for analysis of Cr (VI) solubilized in the alkaline digestate. The hexavalent chromium is determined colorimetrically (Method 7196), by reaction with diphenylcarbazide in acid solution. A red – violet color of unknown composition is produced and the absorbance is measured spectrophotometrically at 540nm.

### **3.0 DEFINITIONS**

- 3.1 BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.
- 3.2 CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of approximately 10 percent. (For some methods this is mandatory and for some it is a

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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recommended only. Refer to individual method SOP's). For most methods, the mid-level calibration check standard criteria is  $\pm 10$  percent of the true value. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

- 3.3 **EXTERNAL CHECK STANDARD.** The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run immediately after calibration. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.
- 3.4 **SPIKE BLANK OR LAB CONTROL SAMPLE.** Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.
- 3.5 **MATRIX:** The component or substrate (e.g., water, soil) which contains the analyte of interest.
- 3.6 **MATRIX DUPLICATE:** A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of  $\pm$  the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of  $\pm 20\%$  RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

- 3.7 **MATRIX SPIKE:** The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result}) \times 100}{(\text{Amount Spiked})} = \text{Matrix Spike Recovery}$$

- 3.8 **METHOD BLANK.** The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less than the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.
- 3.9 **METHOD DETECTION LIMITS (MDLS).** The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLS should be determined approximately once per year for frequently analyzed parameters.
- 3.10 **REAGENT BLANK:** The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.
- 3.11 **REAGENT GRADE:** Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
- 3.12 **REAGENT WATER:** Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.
- 3.13 **REFERENCE MATERIAL:** A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.
- 3.14 **STANDARD CURVE:** A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

**4.1 INTERFERENCES**

- 4.2 For waste materials or soils containing soluble Cr (III) concentrations greater than four times the laboratory Cr (VI) reporting limit, Cr (VI) results obtained using this method may be biased high due to method-induced oxidation. The addition of  $Mg^{2+}$  in a phosphate buffer to the alkaline extraction solution has been shown to suppress this oxidation.
- 4.3 The reaction with diphenylcarbazide is nearly specific for chromium. Hexavalent molybdenum and mercury salts will react to form color with the reagent but the intensities are much lower than that of chromium at the specified pH. Concentrations of molybdenum and mercury (>200 mg/l) can interfere. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.
- 4.4 Iron in concentrations greater than 1 mg/L may produce a yellow color, but the ferric iron color is not strong and difficulty is not normally encountered if absorbance is measured by spectrophotometer at the appropriate wavelength.
- 4.5 For turbid samples or colored samples, prepare a two aliquot of samples. Treat one with no color reagent and another with color reagent. This way will help to eliminate false positive reading due to the nature color of the matrix.

**5.0 SAFETY**

- 5.1 The analyst must follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.
- 5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.
- 5.3 The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: hexavalent chromium.

**6.0 EQUIPMENT AND SUPPLIES**

- 6.1 Spectrophotometer – HACH 2500 DR, for use at 540 nm, a light path of 1 cm or longer.
- 6.1.1 10 ml sample cell, P/N 21228-00

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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- 6.2 Volumetric flasks and pipets and graduated cylinders, class A. All glassware should be washed with soap and tap water and then well rinsed with deionized water.
- 6.3 50 and 250 ml glass beakers with watch glasses. All glassware should be washed with soap and tap water and then well rinsed with deionized water.
- 6.4 Filter paper, 0.45 um. Acceptable filter papers include the following: MSI cellulostic white grid filters, 0.45 um, 47 mm
- 6.5 Filter pump and vacuum filtration apparatus.
- 6.6 pH meter.
- 6.7 Hot plate, capable of maintaining the digestion solutions at 90 to 95 C, with constant stirring ability.
- 6.8 Four place analytical balance.
- 6.9 Thermometer, calibrated to a NIST certified thermometer
- 6.10 Centrifuge Tubes.
- 6.11 One or two place balance.

**7.0 REAGENTS AND STANDARDS**

- 7.1 All reagents should be made from ACS grade reagents unless otherwise noted. Deionized water should be used whenever water is needed. The expiration date for standards and reagents is the date supplied by the manufacturer or if no expiration date is given, a default of 6 months is used. For acid solutions (nitric, sulfuric, hydrochloric) the expiration date is 2 years from the date of preparation of the solution.
- 7.2 Nitric acid, HNO<sub>3</sub>, concentrated, trace metals grade.
- 7.3 Nitric acid, HNO<sub>3</sub>, 5.0 M, trace metals grade. Add 32 ml of concentrated nitric acid to approximately 50 ml of DI water. Dilute to a final volume of 100 ml with DI water and mix well. Store at 20-25°C in the dark. Do not use concentrated nitric acid to make up the 5.0M solution if it has a yellow tinge. The yellow color is indicative of a photo reduction of nitrate to nitrite, a reducing agent for Cr(VI).
- 7.4 Sodium Carbonate, Na<sub>2</sub>CO<sub>3</sub>, anhydrous.
- 7.5 Sodium Hydroxide, NaOH.
- 7.6 Magnesium Chloride, MgCl<sub>2</sub> (anhydrous). Note: 392.18 mg of MgCl<sub>2</sub> is equivalent to 100 mg of Mg<sup>2+</sup>.

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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- 7.7 Phosphate Buffer Solution (0.5 M  $K_2HPO_4$ /0.5 M  $KH_2PO_4$  buffer at pH 7): Dissolve 87.09 g of  $K_2HPO_4$  and 68.04 g of  $KH_2PO_4$  into 700 ml of distilled deionized water. Transfer to a 1 liter volumetric flask and dilute to volume.
- 7.8 Digestion Solution: Dissolve 20.0 g of NaOH and 30.0 g of  $Na_2CO_3$  in distilled deionized water in a one-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20 to 25°C and prepare fresh monthly. The pH of this digestion solution must be checked before using. If the pH is not greater than or equal to 11.5, then the digestion solution should be discarded and a new solution should be made up.
- 7.9 Insoluble hexavalent chromium spike, lead chromate,  $PbCrO_4$ . The insoluble matrix spike is prepared by adding 10 to 20 mg of  $PbCrO_4$  to the insoluble matrix spike aliquot.
- 7.10 Soluble hexavalent chromium spiking solution stock. A 1000 mg/l stock solution of potassium dichromate can be used as the stock solution for the spiking solution (1000 mg/l chromium solution, HACH, P/N 14664-42).
- 7.10.1 Soluble hexavalent chromium spiking solution, 100 mg/l. Add 10.0 ml of the 1000 mg/l hexavalent chromium to a 100 ml volumetric flask and dilute to volume with DI water. Mix well. One (1.00) ml of this spiking solution can be used to spike the soluble matrix spike aliquot. The approximate level of the spike in the spiked sample will be 40 mg/kg.
- 7.11 Sulfuric acid, 10 percent (v/v). Add 10 ml of concentrated sulfuric acid to approximately 70 ml of DI water. Mix well and let cool. Dilute to a final volume of 100 ml with DI water.
- 7.12 Acetone. Do not use acetone that comes in a container with a metal or metal lined cap.
- 7.13 Diphenylcarbazide solution. Dissolve 0.250 g of 1,5 diphenylcarbazide in 50 ml of acetone. Store in a brown or a foil covered bottle to minimize exposure to light. Discard when the solution becomes discolored or monthly, whichever comes first. (Note: Be sure to check the quality of the diphenylcarbazide solution before adding it to the sample.
- 7.14 Hexavalent Chromium Calibration Standard Solutions. The calibration standards must be prepared fresh daily or each time the analysis is run. For instrument calibration, prepare the standards from the stocks as shown below. For all standards, add 50 ml of digestion solution to a labeled plastic beaker and then pipet in the appropriate amount of a stock solution. Do not dilute these standards to the final volume at this time. Refer to step 10.8 in the procedure section for further instructions.
- 7.15 Hexavalent Chromium, 10 mg/l stock solution. Add 1.00 ml of 1000 mg/l hexavalent chromium to a 100 ml volumetric flask and dilute to volume with DI water. Mix well.
- 7.15.1 Add the amount of stock specified below to the 50 ml of digestion solution.
- Blank: No spike is added to the blank.
- |             |                           |
|-------------|---------------------------|
| 0.010 mg/l: | Add 0.10 ml of 10.0 mg/l  |
| 0.025 mg/l: | Add 0.25 ml of 10.0 mg/l. |
| 0.100 mg/l: | Add 1.00 ml of 10.0 mg/l. |

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

0.250 mg/l: Add 2.50 ml of 10.0 mg/l.  
 0.500 mg/l: Add 5.00 ml of 10.0 mg/l

7.15.2 Place a stirring bar in the sample and place it on a stirring plate. Adjust the pH of the solution between 7.00 and 8.00 by carefully adding 5.0 M nitric acid to the digestate while constantly measuring the pH. Do **not** let the pH of the solution go below 7.00. If the pH goes below 7.00, then the digestate must be discarded and a new digestate prepared. Make sure to record the final pH.

7.15.3 Dilute to 100 ml with DI water.

7.16 Hexavalent Chromium, ICV (Initial Calibration Verification) Solutions. The check standards must be prepared fresh daily or each time the analysis is run. Prepare the standards from the stocks as shown below. All check standards must go through the entire digestion process, starting at step 10.3. A minimum of 4 check standards should be made for a batch of 20 samples. Note: The check standards must be made from a different source than the calibration standards.

7.16.1 Hexavalent Chromium 1000 mg/l stock solution. (Absolute Standards P/N 54161)

7.16.2 Hexavalent Chromium, 10 mg/l stock solution. Add 1.00 ml of 1000 mg/l hexavalent chromium to a 100 ml volumetric flask and dilute to volume with DI water. Mix well.

7.16.3 For initial calibration curves that have the 0.500 mg/l standard as the upper limit, a calibration check at 0.250 mg/l must be used. Therefore, add the amount of stock solution specified below to 50 ml of digestion solution. Do not dilute to a final volume. This entire solution should be digested.

7.16.3.1 For 0.250 mg/l: Add 2.5 ml of the 10 mg/l stock solution

**8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE**

8.1 Soil samples should be collected with non-metallic devices and stored field-moist at  $\leq 6^{\circ}\text{C}$  until analysis. Hexavalent chromium has been shown to be quantitatively stable in field-moist soil samples for 30 days from sample collection.

8.2 The alkaline digestate is stable for up to 168 hours after extraction from soil.

**9.0 QUALITY CONTROL**

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements. All quality control data should be maintained and available for easy reference or inspection.

9.1 It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements. It is the responsibility of the analyst to maintain quality control data to confirm acceptable instrument performance and support analytical results reported.

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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- 9.1.1 An initial demonstration of capability (IDOC) must be performed by each analyst. This is the initial analysis of four aliquots at the mid-range standard processed through the entire preparation and analytical method. Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviation of the of the population sample (n-1) (in the same units) for each parameter of interest.
- 9.1.2 An on-going DOC is maintained and demonstrated at least annually through the analysis of single-blind samples (PT sample), or use of four consecutive laboratory control samples compared to the predetermined acceptance limits for precision and accuracy.
- 9.1.3 Method Detection Limit Study: MDL determination performed annually or when major system change. Analyze 7 replicate at concentration 2-5 times of the estimated MDL. Previous MDL values may be used to help determine appropriate spiking levels. Where no previous MDL values exist, reporting limit (RL) may be used as guideline for determining spiking level(s). Refer to ANC SOP-QA006 MDL Determination. The analyst should determine MDLs initially, when any change is made which could affect the MDLs or more frequently if required by the method.
  - 9.1.3.1 In addition, the analyst must demonstrate low level capability on an ongoing basis through an LOD and LOQ determination or repeated low level analyses at the MRL on each instrument for each method, matrix and analyte
  - 9.1.3.2 After each detection limit determination, the laboratory must immediately establish the LOD by spiking a quality system matrix at approximately two to three times the detection limit (for single analyte standard) or one to four times the detection limit (for multi-analyte standard). This spike concentration establishes the LOD. The LOD must be verified quarterly for DoD project.
  - 9.1.3.3 The LOQ must be set within the calibration range prior to sample analysis. At a minimum, the LOQ must be verified quarterly for DoD project.
- 9.2 Analytical Run QC requirement, frequency, acceptance criteria and corrective action to be taken. This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements as taken from these methods. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.
  - 9.2.1 A new 5 point calibration curve must be analyzed on each analysis day. The calibration curve must have a correlation coefficient greater than or equal to 0.995 percent.
  - 9.2.2 An initial calibration verification (ICV) standard at approximately the midpoint of the curve must be analyzed after the calibration curve. The ICV standard must be prepared from different stock than the calibration curve and should be taken through the digestion process as outlined in the procedure section of this SOP.

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- 9.2.3 All samples should initially be analyzed undiluted. If the sample concentration is higher than the highest standard, then the sample should be diluted and reanalyzed. The dilution should be made so that, if possible, the sample is in the mid-range of the calibration curve.
- 9.2.4 One preparation blank is required for each set of 20 samples or less or with each batch, whichever is most frequent. The preparation blank must contain all of the reagents in the sample volumes as used in the preparation of the samples. (The preparation blank should never be used to blank correct the samples.) The preparation blank must be less than the reporting limit. If the preparation blank does not meet the criteria, then the entire batch must be redigested.
- 9.2.5 A continuing calibration verification (CCV) standard at approximately the mid-point of the curve must be analyzed after every 10 samples or every 20 readings (10 sample readings plus 10 background readings). All CCV standards must be within 10 percent of the true value for that standard. If they are outside of this range, do not proceed. Check with the laboratory supervisor or manager for further directions.
- 9.2.6 A reagent blank (or CCB) must be analyzed after each CCV. The reagent blank must be less than the reporting detection limit. If this criteria is not met do not proceed. Check with the laboratory supervisor or manager for further directions.
- 9.2.7 A duplicate sample must be prepared and analyzed for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. Acceptance criteria of 20 percent relative percent difference should be applied if the original and duplicate sample values are greater than or equal to 4 times the reporting detection limit. If the values are less than 4 times the reporting detection limit, then a control limit of  $\pm$  the reporting detection limit should be applied.
- 9.2.8 Both a soluble and an insoluble hexavalent chromium matrix spike must be prepared and analyzed for each set of 20 samples of a similar matrix. The acceptance range for matrix spike recoveries is 75 to 125 percent recovery. If the matrix spikes recoveries for either the soluble or the insoluble spikes are not within these recovery limits, then the lab supervisor or manager must be immediately notified. The client services department will then be notified to contact the client. The method requires additional testing as listed below, but the lab should not proceed with this testing until client approval is obtained and the testing is logged into the LIMS system.
- 9.2.8.1 All samples and quality control must be rehomogenized, redigested and reanalyzed to verify the original sample results.
- 9.2.8.2 Additional tests, such as oxidation-reduction potential, pH, sulfide, ferrous iron, etc., may be requested to help quantify the reducing nature of the sample. For some projects, eH and pH analysis may be specified for all samples at the start of the project. Eh and pH data plots must be provided in the data deliverable if this analysis is specified

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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A value of Eh-pH below the bold diagonal line on attachment (Figure 2-Eh/pH phase diagram) indicates a reducing soil for Cr (VI). The downward slope to the right indicates that the Eh value, at which Cr (VI) is expected to be reduced, decreases with increasing pH. The presence of H<sub>2</sub>S or other strong odors indicates a reducing environment for Cr (VI). In general, acidic conditions accelerate reduction of Cr (VI) in soils, and alkaline conditions tend to stabilize Cr (VI) against reduction. If pre-digestion matrix spike recovery is not within the recovery limits, the reductive nature of the sample must be documented. This is done by plotting the Eh and pH data on the Eh-pH diagram (See attachment) to see if spike recovery is or is not expected in the soil. If the data point falls below the Cr (VI)-Cr (III) line on the diagram, then the data is not qualified or rejected. The sample is reducing for Cr (VI). If the data point falls above the line, then the sample is capable of supporting Cr (VI). In this case, technical error may be responsible for the poor spike recovery, and the extraction should be repeated, along with the Eh and pH measurements. If re-extraction results in a poor spike recovery again, then the data is qualified. At this point, review of other soil characteristics, such as levels of pH, Eh, TOC, sulfides, Fe (II), is appropriate to understand why poor spike recovery occurred. This extra review of these soil properties is only necessary if the unspiked sample contains detectable Cr (VI).

A mass balance study for total chromium may be done, using the digested solids remaining after the alkaline digestion and filtration of the matrix spike and from an unspiked aliquot of the sample.

- 9.2.9 A post-digest spike must be prepared and analyzed for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. The acceptance range for post digest spike recoveries is 85 to 115 percent recovery.
- 9.2.10 Spike blank or lab control sample must be prepared and analyzed for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. The spike blank or lab control can be prepared using either soluble or insoluble hexavalent chromium as the spike. The acceptance range for the spike blanks is 80 to 120 percent recovery. If the spike blanks are not within that range, then the entire batch must be redigested and reanalyzed.

**10.0 DIGESTION PROCEDURE**

Below is a step-by-step procedure for the digestion of samples for the determination of hexavalent chromium.

- 10.1 For each sample to be analyzed, weight out  $2.5 \pm 0.10$  g of the sample into a clean, labeled glass beaker. A one or two place balance may be used for this weighing. The sample should be well mixed before the aliquot is removed as described in QA026-0, the representative sample aliquot SOP.
- 10.1.1 For the sample that is to be used for the quality control sample, weigh out six  $2.5 \pm 0.10$  g aliquots from the well mixed sample. One aliquot will be for the soluble Cr(VI) matrix spike, one aliquot will be for the insoluble Cr(VI) matrix spike, one aliquot will be for the original sample analysis, one aliquot will be for the duplicate sample analysis, and the remaining two

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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aliquots will be used to complete the procedures required if the initial post-digest spike does not meet the  $\pm 15$  criteria.

- 10.2 Add the spikes to the matrix spikes and the spike blanks.
  - 10.2.1 Spike the soluble Cr (VI) matrix spike with 1.0 ml of the 100 mg/l Cr(VI) spiking solution. (S1).
  - 10.2.2 Spike the soluble Cr(VI) spike blank with 1.0 ml of the 100 mg/l Cr(VI) spiking solution.(B1)
  - 10.2.3 Using an analytical balance, weigh out 0.010 to 0.020 g of  $PbCrO_4$  onto a clean piece of weighing paper and carefully add the spike into the insoluble matrix spike sample. Make sure to record the weight used. (S3)
  - 10.2.4 Using an analytical balance, weigh out 0.010 to 0.020 g of  $PbCrO_4$  onto a clean piece of weighing paper and carefully add the spike into the insoluble spike blank sample. Make sure to record the weight used. (B2)
  - 10.2.5 Spike the soluble Cr Vi spike blank with 3.75 ml of the 100 mg/L Cr Vi spiking solution.(S2)
- 10.3 Add 50 ml of digestion solution to each sample. Also add 0.392 g of  $MgCl_2$  (from 0.38 to 0.42g) and 0.5 ml of the 1.0 M phosphate buffer. In addition to the samples, 3 extra beakers should be prepared for the method blank, the soluble Cr(VI) spike blank, and the insoluble Cr(VI) spike blank.
- 10.4 In addition to the samples, the ICV (initial calibration check) standards should also be digested. Add the entire ICV solution (refer to step 9.16) into a clean, labeled glass beaker. Also add approximately 0.392 g of  $MgCl_2$  and 0.5 ml of the 1.0 M phosphate buffer.
- 10.5 Cover all samples and quality control (including the calibration check samples) with watch glasses. Add a stirring bar to each sample and stir the samples for at least 5 minutes without heating.
- 10.6 Place the samples on a stirring hot plate that has been preheated to 90 to 95°C. Heat the samples with constant stirring for 60 minutes, maintaining a temperature range of 90 to 95°C. The temperature should be measured by placing a calibrated thermometer in an extra beaker containing digestion reagent on the hot plate. The temperature must be recorded at 30 minutes and 60 minutes during the digestion process. Both the start and the stop time of the digestion must be recorded.
- 10.7 Cool the samples to room temperature. Filter them through 0.45  $\mu m$  filter paper. Rinse the filter and filtration apparatus with DI water and transfer the filtrate into labeled graduated plastic beakers.
  - 10.7.1 If the filters become clogged using the 0.45  $\mu m$  filter paper, a larger size filter paper (Whatman GFB or GFF) may be used to prefilter the samples. However, the final filtration must be through the 0.45  $\mu m$  filters. If a pre-filtration is required, it should be recorded on the digestion log.
  - 10.7.2 The solids and the filter remaining after the filtration of the matrix spikes may need to be saved in a labeled plastic beaker and stored in the refrigerator. If low recoveries are

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obtained on the matrix spikes, these solids may be needed for additional analyses. Check with the area supervisor or manager for further instructions.

- 10.7.3 At this point, the digestates are stable and may be held for up to 7 days hours before proceeding with step 10.8.
- 10.8 Do not start this step unless the analysis will be started within one hour after this step has been completed. The calibration standards should also be taken through this process.
- 10.8.1 Place a stirring bar in the sample and place it on a stirring plate. Adjust the pH of the solution between 7.00 and 8.00 by carefully adding 5.0 M nitric acid to the digestate while constantly measuring the pH. Do not let the pH of the solution go below 7.00. If the pH goes below 7.00, then the digestate must be discarded and a new digestate prepared. Make sure to record the final pH.
- 10.8.2 If the pH is changing too rapidly with 5.0 M nitric acid, then a more dilute solution of nitric acid may be used for the pH adjustment.
- 10.8.3 Carbon dioxide and nitric acid fumes will be evolved during this process. Therefore, this step must be performed in a hood or well ventilated area.
- 10.9 Quantitatively transfer the contents of the beaker to a 100 ml volumetric flask or class A graduated cylinder and adjust the sample volume to the mark with DI water. Mix well. At this point, a brief description of each sample (color, turbidity, etc.) can be added to the digestion log.
- 10.9.1 If the same cylinder is used for multiple samples, it must be rinsed with deionized water at least 3 times between samples.

**11.0 ANALYSIS PROCEDURE**

- 11.1 Turn on the spectrophotometer and let it warm up for at least 30 minutes. Set the wavelength to 540 nm and adjust the zero.
- 11.2 Using a class A graduated cylinder, transfer quantitatively 45.0 ml of the sample or standard to be analyzed to centrifuge tube.
- 11.3 Add 1.0 ml of diphenylcarbazide solution and mix well.
- 11.4 Slowly add 10 percent sulfuric acid to each sample, mixing well after each addition. Adjust the pH to a range of 1.5 to 2.5. Test the pH of each sample with a pH meter when the effervescence is minimal and record this reading. (On some samples, a small amount of effervescence has been observed several hours after the pH adjustment was completed.)
- 11.4.1 A background correction point must also be prepared for each sample with 10 ml of sample adjusted to a pH of 1.5 to 2.5 with sulfuric acid. The background correction point should not contain diphenylcarbazide. Make sure to record the adjusted pH of the background correction point.

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- 11.5 If the samples are turbid at this point, filter them through a 0.45 um filter. If the sample aliquot is filtered, the background aliquot must also be filtered.
- 11.6 Transfer the samples to 50 ml volumetric flasks or class A graduated cylinders and dilute to a final volume of 50 ml with DI water. Let the samples stand for 5 to 10 minutes after the reagents are added for full color development.
- 11.6.1 If the same cylinder is used for multiple samples, it must be rinsed with deionized water at least 3 times between samples.
- 11.7 Read the standard calibration curve first, and then a calibration check standard and a reagent blank, making sure to record all results on the strip chart recorder. The correlation coefficient for the curve must be greater than or equal to 0.995, the check standard must be within 10 percent of the true value, and the reagent blank must be less than the reporting detection limit before the analysis can be continued.

$$r = \frac{\sum(x - \bar{x})(y - \bar{y})}{\sqrt{\sum(x - \bar{x})^2 \sum(y - \bar{y})^2}}$$

Where r = correlation coefficient  
 x = amount of analyte  
 y = response of instrument  
 $\bar{x}$  = average of x values  
 $\bar{y}$  = average of y values

- 11.8 After the curve and the initial quality control are completed, the samples may be analyzed. First read the sample result. If the result is over the highest point in the calibration curve, do not read the background correction point. If the result is within the calibration curve, the background correction point must be read immediately after the sample analysis is complete and before starting the next sample.
- 11.9 After every 10 samples or every 20 readings (10 samples plus 10 background correction points), a CCV and a reagent blank will be analyzed. The reagent blank must be less than the reporting limit, and the CCV must be within 10% of the true value. If they are outside of this range, do not proceed. Check with the laboratory supervisor or manager for further directions.
- 11.10 After the quality control sample analysis is completed, prepare a post-digest spike on this sample. The sample should be spiked at 2 times the concentration found in the original sample aliquot or 40 mg Cr(VI)/kg, whichever is greater. Then proceed through steps 11.3 to 11.6 and analyze the spiked sample. Calculate the recovery immediately. If the recovery is not within 85 to 115 percent, proceed to steps 11.11 and 11.12.
- 11.10.1 The 40 mg/kg spike can be made by spiking a 45 ml aliquot of digestate containing 1.125 g of digested sample with 0.45 ml of 100 mg/l Cr(VI) standard (Section 7.11).

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

11.10.2 This spiking level requirement is taken from method 3060a. A lower level spiking requirement is given in the NJDEP 7196A method, but guidance from the state suggested using the 3060A spiking levels when following the 3060a digestion.

- 11.11 Dilute by a factor of 1:5 a fraction of the quality control sample. Place 45 ml of the diluted sample into a centrifuge tube. Then proceed through steps 11.3 to 11.6 and analyze the sample. Also prepare a background correction point at this dilution and analyze it immediately following the analysis of the diluted sample.
- 11.12 Take an additional 45 ml aliquot of the sample and adjust the pH to between 8.0 and 8.5 using 1.0 N NaOH. Record the final pH. Then spike the sample at 2 times the concentration found in the original sample aliquot or 40 mg Cr(VI)/kg, whichever is greater. After the sample is spiked, proceed with steps 11.3 through 11.6, and analyze the pH adjusted post-digest spike.
- 11.13 The calculations should be done as shown below. Values less than the IDL should be treated as zero for all calculations.

11.13.1 Calculation of the sample result.

Conc. Cr (VI) in the sample in mg/kg =

$$\frac{(\text{Conc. in digestate in ug/ml}) \times (\text{final volume in ml}) \times \text{DF}}{(\text{Initial sample weight in g}) \times (\% \text{solids}/100)}$$

11.13.2 Calculation of amount spiked.

Spike amount (SA) in mg/kg =

$$\frac{(\text{Conc. of spiking solution, ug/ml}) \times (\text{vol. of spike, ml})}{(\text{Initial sample weight in g}) \times (\% \text{solids}/100)}$$

11.13.3 Calculation of matrix spike recovery.

$$\text{MS Rec.} = \frac{(\text{SSR} - \text{SR}) \times 100}{\text{SA}}$$

Where SSR = Spiked sample result  
 SR = Sample result and  
 SA = Spike added.

11.13.4 Calculation of duplicate rpd.

$$\text{Dup RPD.} = \frac{(\text{SR} - \text{DR}) \times 100}{\{( \text{SR} + \text{DR} ) / 2 \}}$$

where SR = Sample result and  
 DR = Duplicate result.

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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**12.0 DATA ANALYSIS AND CALCULATIONS**

- 12.1 All samples should be updated to analysis (GN) batches in the LIMS system. The analyst should calculate all matrix spike, duplicate, external, and CCV recoveries and review the results of all blanks.
- 12.2 All documentation must be completed, including reagent references and spike amounts and spiking solution references.
- 12.3 A data file should be exported to the LIMS system and the spike amounts should be entered into the file at the GNAPP process step.
- 12.4 A final data package, consisting of the prep and analysis raw data, the LIMS cover page, the reagent reference pages, and the QC summary pages must be turned into the area supervisor or other senior reviewer for review.
- 12.5 After review by the supervisor, the data is released in the LIMS for access to the clients.

**13.0 POLLUTION PREVENTION**

- 13.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed.

**14.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QC MEASURES**

- 14.1 All methods will refer to Section 9.0

**15.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA**

- 15.1 All method will refer to Section 9.0

**16.0 CONTINGENCIES OF HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA**

- 16.1 All contingencies for Out of Control and/or Unacceptable data require the approval of Laboratory Director and/or, QA Officer.

**17.1 WASTE MANAGEMENT** –All methods will refer to the Waste Handling SOP or Safety Manual

- 17.2 Non hazardous aqueous wastes.
- 17.3 Hazardous aqueous wastes.

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**Hexavalent Chromium in Soils by Alkaline Digestion SW846 Method 3060A and 7196A**

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- 17.4 Chlorinated organic solvents.
- 17.5 Non-chlorinated organic solvents.
- 17.6 Hazardous solid wastes.

**18.0 METHOD PERFORMANCE**

- 18.1 All methods will refer to the published method for performance data.

**19.0 REFERENCES**

- 19.1 EPA SW-846, Method 3060A "Alkaline Digestion for hexavalent chromium", Revision 1 December 1996
- 19.2 EPA SW-846, Method 7196A "Chromium, Hexavalent (Colorimetric)", Revision 1 July 1992
- 19.3 HACH Oddysey DR/2500 manual

**20.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA**

- 20.1 All methods shall refer to the published method for the Tables, Diagrams, Flow charts, and Validation data.

# Quality Systems Manual

Revision XI: September, 2012

Effective Date: September 4, 2012

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Approved by: (Signature/Date)	
<b>Dr. Kesavalu Bagawandoss</b> Laboratory / Technical Director	See Hardcopy for Signature
<b>Guergana Gueorguieva</b> Quality Assurance Officer	See Hardcopy for Signature

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Introduction

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Revision Date: September 4, 2012

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The Accutest Laboratories – Northern California Quality Assurance System, detailed in this plan, has been designed to meet the quality program requirements of the National Environmental Laboratory Accreditation Conference (NELAC), ISO Guide 17025, ISO Guide 17011 and other National environmental monitoring programs. The plan establishes the framework for documenting the requirements of the quality processes regularly practiced by the Laboratory. The Quality Assurance Director is responsible for changes to the Quality Assurance Program, which is appended to the Quality System Manual (QSM) during the annual program review. The plan is also reviewed annually for compliance purposes by the Company President and Laboratory Director and edited if necessary. Changes that are incorporated into the plan are itemized in a summary of changes following the introduction. Plan changes are communicated to the general staff in a meeting conducted by the Director of Quality Assurance following the plan's approval.

The Accutest plan is supported by standard operating procedures (SOPs), which provide specific operational instructions on the execution of each quality element and assure that compliance with the requirements of the plan are achieved. Accutest employees are responsible for knowing the requirements of the SOPs and applying them in the daily execution of their duties. These documents are updated as changes occur and the staff is trained to apply the changes.

At Accutest, we believe that satisfying client requirements and providing a product that meets or exceeds the standards of the industry is the key to a good business relationship. However, client satisfaction cannot be guaranteed unless there is a system that assures the product consistently meets its design requirements and is adequately documented to assure that all procedural steps are executed, properly documented and traceable.

This plan has been designed to assure that this goal is consistently achieved and the Accutest product withstands the rigors of scrutiny that are routinely applied to analytical data and the processes that support its generation.

Revision Date: September 4, 2012

**Summary of Changes**  
**Accutest Laboratories Quality System Manual – February 2011**

<b>Section</b>	<b>Description</b>
-----	SOP Number, Updated Revision Number and Date
	Original issue due to Sale February 2008
	<b>QA008_2 - Revision II May 1, 2008 Changes</b>
Appendix II	Updated SOP List
2.3	Assigned Deputies to Laboratory Director and Quality Assurance Officer
	<b>QA008_3 - Revision III March 6, 2009 Changes</b>
Appendix IV	Updated Equipment list – add ICS2000
Sect. 2	Updated Organizational Chart
	<b>QA008_4 - Revision IV September 7, 2009 Changes</b>
Cover page	Change address
Section 1.1	Added including the DoD QSM
Section 2.1	Change Santa Clara to San Jose
Section 2.1	Added ANC is a permanent location and not a mobile of temporary facility
Section 2.3	Added the laboratory designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function when the technical director is absent for a period of time exceeding 15 days. If this absence exceeds 65 consecutive days, the primary accrediting authority shall be notified in writing
Section 6.1	Standard Operating Procedures (SOP), deleted in the master SOP binder
Section 9.8	Subcontract Laboratory Evaluation-added All subcontracted items for testing covered under DoD will only be submitted to DoD-ELAP laboratories
Section 15.1	Procedure-inserted If no systematic defects are present and the proposed resolutions is sufficient, QA will close the complaint/inquiry with a No Further Action is necessary
	<b>QA008_5- Revision V December 31, 2009 changes</b>
Cover, 2.2,2.3, 3.1,4.1,5.1	Changing the title and responsibilities from “Laboratory Director” to “Laboratory/Technical Director”.
5.1	Signatory Hierarchy Changed
	<b>QA008_6- Revision VI February 15, 2010 changes</b>
5.1	Signatory Hierarchy Changed
2.1,2.2	Added Accutest Laboratories-Mountain States to the Network.
10.4	Added text to define a Second Source Standard.
	<b>QA008_7- Revision VII October 20, 2010 changes</b>
Section 2.3	Accutest Northern California – Organization Chart
Appendix II	Standard Operational Procedures Directory
Appendix IV	Laboratory Equipment
	<b>QA008_8-Revision VIII February 18, 2011 changes</b>
Section 2.3	Accutest Northern California – Organization Chart
Section 16.3	Added Data Inquiry Program
Appendix II	Standard Operating Procedures
Appendix IV	Laboratory Equipment –added GCMS Q, GCMS R and Thermo Fisher ICP
	<b>QA008_9-Revision IX August 3, 2011 changes</b>
Cover, Section 2.3	Accutest Northern California – Organization Chart
	<b>QA008_10-Revision X May 1, 2012 changes</b>
Section 2.3	Accutest Northern California – Organization Chart
Appendix II, IV	Standard Operating Procedures,Laboratory Equipment
Cover, Section 2.3	<b>QA008_11-Revision XI September 4, 2012 changes-ALNC Org Chart</b>

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## 1.0 QUALITY POLICY

### 1.1 Accutest Mission

Accutest Laboratories provides analytical services to commercial and government clients in support of environmental monitoring and remedial activities as requested. The Laboratory's mission is dedicated to providing reliable data that satisfies client's requirements as explained in the following:

***“Provide easy access, high quality, analytical support to commercial and government clients including those following the DoD QSM, which meets or exceeds data quality objectives and provides them with the data needed to satisfy regulatory requirements and/or make confident decisions on the effectiveness of remedial activities.”***

These services are provided impartially and are not influenced by undue commercial or financial pressures which might impact the staff's technical judgment. Coincidentally, Accutest does not engage in activities that endanger the trust in our independent judgment and integrity in relation to the testing activities performed.

### 1.2 Policy Statement

*The management and staff of Accutest Laboratories share the responsibility for product quality. Accordingly, ANC's quality assurance program is designed to assure that all processes and procedures, which are components of environmental data production, meet established industry requirements, are adequately documented from a procedural and data traceability perspective, and are consistently executed by the staff. It also assures that analytical data of known quality, meeting the quality objectives of the analytical method in use and the data user's requirements, is consistently produced in the laboratory. This assurance enables the data user to make rational, confident, cost-effective decisions on the assessment and resolution of environmental issues.*

*The laboratory Quality System also provides the management staff with data quality and operational feedback information. This enables them to determine if the laboratory is achieving the established quality and operational standards, which are dictated by the client or established by regulation. The information provided to management, through the QA program, is used to assess operational performance from a quality perspective and to perform corrective action as necessary.*

*All employees of Accutest Laboratories participating in environmental testing receive quality system training and are responsible for knowing and complying with the system requirements. The entire staff shares ANC's commitment to good professional practice.*

Phillip B. Rooney  
Chairman and CEO

May 01, 2012

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Date

## 2.0 ORGANIZATION

2.1 ***Organizational Entity.*** Accutest Laboratories is a privately held, independent testing laboratory founded in 1956 and registered as a New Jersey Corporation. The headquarters are located in Dayton, New Jersey where it has conducted business since 1987. Satellite laboratories are maintained in Marlborough, Massachusetts; Orlando, Florida; Houston, Texas; San Jose, California and Wheatridge, Colorado, Lafayette, Louisiana.

***Accutest Northern California is a permanent location and not a mobile or temporary facility***

## 2.2 ***Management Responsibilities***

***Requirement.*** Each laboratory facility has an established chain of command. The duties and responsibilities of the management staff are linked to the President/CEO of Accutest Laboratories who establishes the agenda for all company activities.

**President/CEO.** Primary responsibility for all operations and business activities. Delegates authority to laboratory directors, general managers, and the quality assurance director to conduct day to day operations and execute quality assurance duties. Each of the seven operational entities (New Jersey, Florida, Massachusetts, California, Texas, Louisiana and Colorado) report to the President/CEO.

**Vice President Operations** (*Corporate*). Delegates responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures to the respective Laboratory and/or Technical Directors. VP of Operations reports directly to the President/CEO.

**Laboratory Director** (*Local*). Executes day to day responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures. Reports directly to the Vice President of Operations.

**Corporate Quality Assurance Director** (*Corporate*). Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

**Quality Assurance Officer** (*Local*). Responsible for design support, implementation support, and monitoring support of the quality system. Training personnel in various aspects of quality system. Conducts audits and product reviews to identify product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

**Department Supervisors.** Executes day to day responsibility for specific laboratory areas including technical aspects of production activities and associated logistical procedures. Direct report to the laboratory director.

**Bench Analysts.** Responsible for applying the requirements of the Quality Program to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

### 2.3 *Chain of Command*

The laboratory designates another full-time staff member meeting the qualifications of the technical director(s) to temporarily perform this function when the technical director(s) is absent for a period of time exceeding 15 consecutive calendar days. If this absence exceeds 65 consecutive calendar days, the primary accrediting authority shall be notified in writing.

The responsibility for managing all aspects of ANC's operation is delegated to specific local individuals, who have been assigned the authority to act in the absence of the senior staff. Deputies will perform duties in the absence of their designees if the absence is greater than three weeks. These individuals are identified in the following Chain of Command:

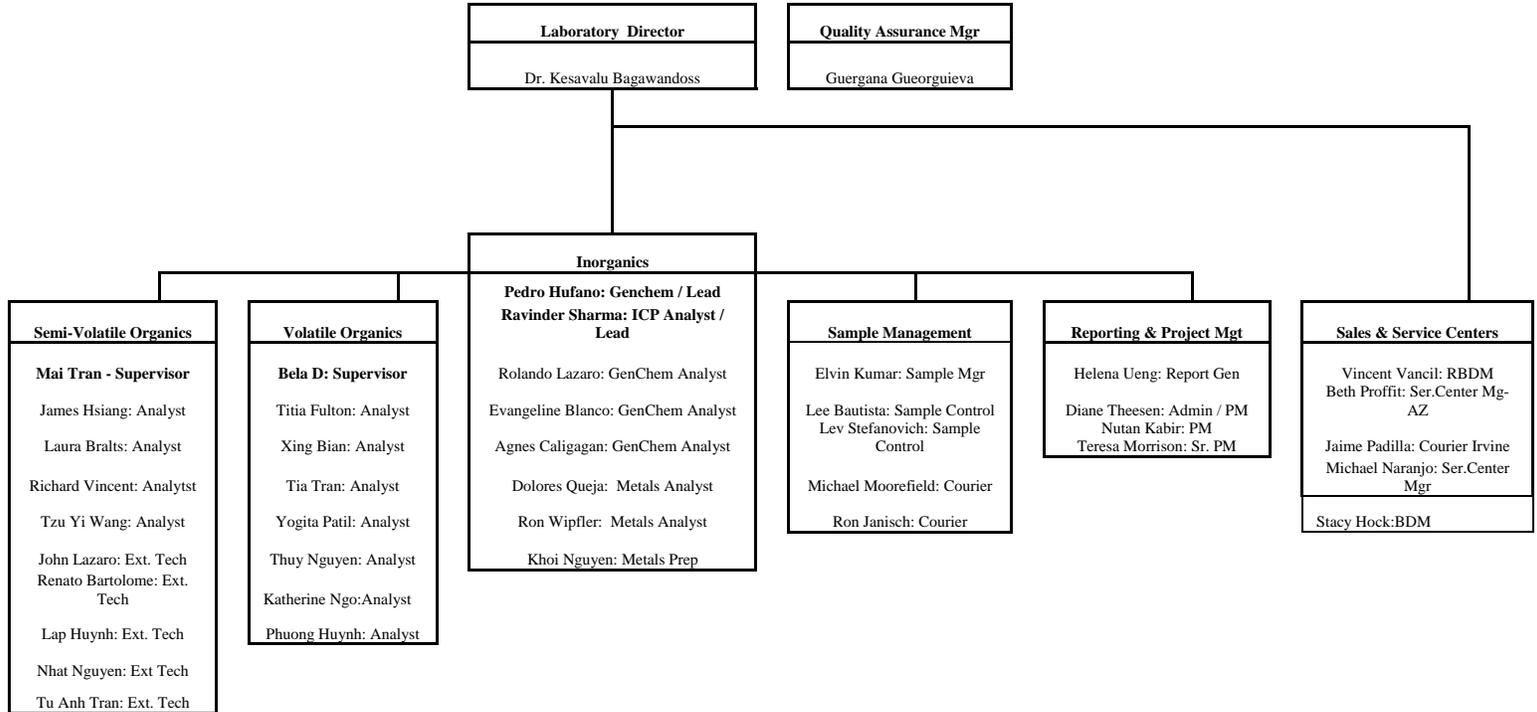
Dr. Kesavalu Bagawandoss; Laboratory/Technical Director

Deputy: Mai Tran

Guergana Gueorguieva; Quality Assurance Officer

Deputy: Kesavalu Bagawandoss

# Accutest Northern California - Organization



### 3.0 QUALITY RESPONSIBILITIES OF THE MANAGEMENT TEAM

3.1 ***Requirement.*** Each member of the management team has a defined responsibility for the Quality System. System implementation and operation is designated as an operational management responsibility. System design and implementation is designated as a Quality Assurance Responsibility.

**President/CEO.** Primary responsibility for all quality activities. Delegates program responsibility to the Quality Assurance Director. Serves as the primary alternate in the absence of the Quality Assurance Director. Has the ultimate responsibility for implementation of the Quality System.

**Vice President Operations (Corporate).** Responsible for implementing and operating the Quality System in all laboratory areas. Responsible for the design and implementation of corrective action for defective processes. Has the authority to delegate Quality System implementation responsibilities.

**Quality Assurance Director (Corporate)** Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if quality issues warrant immediate action. Monitors implemented corrective actions for compliance.

**Quality Assurance Officer (Local).** Responsible for design, implementation support, training, and monitoring support for the quality system. Conducts audits and product reviews to identify product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Provides monitors support for implemented corrective actions for compliance.

**Laboratory/Technical Director.** Responsible for implementing, operating, and technical reviewing of the Quality System in all the local laboratory areas. Responsible for design and implementation of the corrective action process.

**Department Supervisors.** Responsible for applying the requirements of the Quality System in their section and assuring subordinate supervisors and staff apply all system requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

**Bench Analysts.** Responsible for applying the requirements of the Quality System to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

3.2 ***Program Authority.*** Authority for program implementation originates with the President/CEO who bears the ultimate responsibility for system design, implementation, and enforcement of requirements.

This authority and responsibility is delegated to the Director of Quality Assurance who performs quality functions independently without the encumbrances or biases associated with operational or production responsibilities to ensure an honest, independent assessment of quality issues.

- 3.3** ***Data Integrity Policy.*** The Accutest Data Integrity Policy reflects a comprehensive, systematic approach for assuring that data produced by the laboratory accurately reflects the outcome of the tests performed on field samples and has been produced in a bias free environment by ethical professionals. The policy includes a commitment to technical ethics, staff training in ethics and data integrity, an individual attestation to data integrity and procedures for evaluating data integrity. Senior management assumes the responsibility for assuring compliance with all technical ethics elements and operation of all data integrity procedures. The staff is responsible for compliance with the ethical code of conduct and for practicing data integrity procedures.

The Accutest Data Integrity Policy is as follows:

***“Accutest Laboratories is committed to producing data that meets the data integrity requirements of the environmental regulatory community. This commitment is demonstrated through the application of a comprehensive data integrity program that includes ethics and data integrity training, data integrity evaluation procedures, staff participation and management oversight. Adherence to the specifications of the program assures that data provided to our clients is of the highest possible integrity and can be used for decision making processes with high confidence.”***

### **Data Integrity Responsibilities**

***Management.*** Senior management retains oversight responsibility for the data integrity program and retains ultimate responsibility for execution of the data integrity program elements. Senior management is responsible for providing the resources required to conduct ethics training and operate data integrity evaluation procedures. They also include responsibility for creating an environment of trust among the staff and being the lead advocate for promoting the data integrity policy and the importance of technical ethics. The Quality Assurance Director is the designated ethics officer for the Company.

***Staff.*** The staff is responsible for adhering to the company ethics policy as they perform their duties and responsibilities associated with sample analysis and reporting. By executing this responsibility, data produced by Accutest Laboratories retains its high integrity characteristics and withstands the rigors of all data integrity checks. The staff is also responsible for adhering to all laboratory requirements pertaining to manual data edits, data transcription and data traceability. These include the application of approved manual peak integration and documentation procedures. It also includes establishing traceability for all manual results calculations and data edits.

**Ethics Statement.** The Accutest ethics statement reflects the standards that are expected for businesses that provide environmental services to regulated entities and regulatory agencies on a commercial basis. The Ethics Policy is comprised of key elements that are essential to organizations

that perform chemical analysis for a fee. As such, it focuses on elements related to personal, technical and business activities.

Accutest Laboratories provides analytical chemistry services on environmental matters to the regulated community. The data the company produces provides the foundation for determining the risk presented by a chemical pollutant to human health and the environment. The environmental industry is dependent upon the accurate portrayal of environmental chemistry data. This process is reliant upon a high level of scientific and personal ethics.

It is essential to the Company that each employee understands the ethical and quality standards required to work in this industry. Accordingly, Accutest has adopted a code of ethics, which each employee is expected to adhere to as follows:

- o Perform chemical analysis using accepted scientific practices and principles.
- o Perform tasks in an honest, principled and incorruptible manner inspiring peers & subordinates.
- o Maintain professional integrity as an individual.
- o Provide services in a confidential, honest, and forthright manner.
- o Produce results that are accurate and defensible.
- o Report data without any considerations of self-interest.
- o Comply with all pertinent laws and regulations associated with assigned tasks and responsibilities.

**Data Integrity Procedures.** Four key elements comprise the Accutest data integrity system. Procedures have been implemented for conducting data integrity training and for documenting that employees conform to the Accutest Data Integrity and Ethics policy.

The data integrity program consists of routine data integrity evaluation and documentation procedures to periodically monitor and document data integrity. These procedures are documented as SOPs. SOPs are approved and reviewed annually following the procedures employed for all Accutest SOPs. Documentation associated with data integrity evaluations is maintained on file and is available for review.

**Data Integrity Training.** Accutest employees receive technical ethics training during new employee orientation. Employees are also required to refresh their ethical conduct agreement annually, which verifies their understanding of ANC's ethics policy and their ethical responsibilities. A brochure summarizing the details of the Accutest Data Integrity Policy is distributed to all employees with the Ethical Conduct Agreement. The refreshed agreements are archived.

The training focuses on the reasons for technical ethic training, explains the impact of data fraud on human health and the environment, and illustrates the consequences of criminal fraud on businesses and individual careers. ANC's ethics policy and code of ethics are reviewed and explained for each new employee.

Training on data integrity procedures are conducted by individual departments for groups involved in data operations. These include procedures for manual chromatographic peak integration, traceability for manual calculations and data transcription.

**Data Integrity Training Documentation.** Records of all data integrity training are maintained in training folders. Attendance at all training sessions is documented and maintained in the training archive.

**Accutest Data Integrity and Ethical Conduct Agreement.** All employees are required to sign a Data Integrity and Ethical Conduct Agreement annually. This document is archived in training folder, which are retained for duration of employment.

The Data Integrity and Ethical Conduct Agreement is as follows:

- I. *I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at Accutest Laboratories-Northern California.*
- II. *I have received formal instruction on the code of ethics that has been adapted by Accutest Laboratories-Northern California during my orientation and agree to comply with these requirements.*
- III. *I have received formal instruction on the elements of Accutest Laboratories' Data Integrity Policy and have been informed of the following specific procedures:*
  - a. *Formal procedures for the confidential reporting of data integrity issues are available, which can be used by any employee,*
  - b. *A data integrity investigation is conducted when data issues are identified that may negatively impact data integrity.*
  - c. *Routine data integrity monitoring is conducted on sample data, which may include an evaluation of the data I produce,*
- IV. *I have read the brochure detailing Accutest Laboratories Data Integrity and Ethics Program as required.*
- V. *I am aware that data fraud is a punishable crime that may include fines and/or imprisonment upon conviction.*
- VI. *I also agree to the following:*
  - a. *I shall not intentionally report data values, which are not the actual values observed or measured.*
  - b. *I shall not intentionally modify data values unless the modification can be technically justified through a measurable analytical process.*
  - c. *I shall not intentionally report dates and times of data analysis that are not the true and actual times the data analysis was conducted.*
  - d. *I shall not condone any accidental or intentional reporting of inauthentic data by other employees and immediately report it's occurrence to my superiors.*
  - e. *I shall immediately report any accidental reporting of inauthentic data by myself to my superiors.*

**Data Integrity Monitoring.** Documented procedures are employed for performing data integrity monitoring. These include regular data review procedures by supervisory and management staff (Section 12.7), supervisory review and approval of manual integrations and periodic reviews of GALP audit trails from the LIMS and all computer controlled analysis.

*Data Review.* All data produced by the laboratory undergoes several levels of review, which includes two levels of management review. Detected data anomalies that appear to be related to data integrity issues are isolated for further investigation. The investigation is conducted following the procedures described in this section.

*Manual Peak Integration Review and Approval.* Routine data review procedures for all chromatographic processes includes a review of all manual chromatographic peak integrations. This review is performed by the supervisory staff and consists of a review of the machine integration compared to the manual integration. Manual integrations, which have been performed in accordance with ANC's manual peak integration procedures, are approved for further processing and release. Manual integrations which are not performed to ANC's specifications are set aside for corrective action, which may include analyst retraining or further investigation as necessary.

*GALP Audit Trail Review.* Good Automated Laboratory Practice (GALP) audits are comprehensive data package audits that include a review of raw data, process logbooks, processed data reports and GALP audit trails from individual instruments and LIMS. GALP audit trails, which record all electronic data activities, are available for the majority of computerized methodology and the laboratory information management system (LIMS). These audit trails are periodically reviewed to determine if interventions performed by technical staff constitute an appropriate action. The review is performed on a recently completed job and includes interviews with the staff that performed the analysis. Findings indicative of inappropriate interventions or data integrity issues are investigated to determine the cause and the extent of the anomaly.

**Confidential Reporting Of Data Integrity Issues.** Data integrity concerns may be raised by any individual to their supervisor. Employees with data integrity concerns should always discuss those concerns with their immediate supervisors as a first step unless, the employee is concerned with the confidentiality of disclosing data integrity issues or is uncomfortable discussing the issue with their immediate supervisors. The supervisor makes an initial assessment of the situation to determine if the concern is related to a data integrity violation. Those issues that appear to be violations are documented by the supervisor and referred to the Quality Assurance Officer for investigation.

Documented procedures for the confidential reporting of data integrity issues in the laboratory are part of the data integrity policy. These procedures assure that laboratory staff can privately discuss ethical issues or report items of ethical concern without fears of repercussions with senior staff.

Employees with data integrity concerns that they consider to be confidential are directed to the Corporate Human Resources Manager in Dayton, New Jersey. The HR Manager acts as a conduit to arrange a private discussion between the employee and the Corporate QA Director or a local QA Officer.

During the employee - QA discussion, the QA representative evaluates the situation presented by the employee to determine if the issue is a data integrity concern or a legitimate practice. If the practice is legitimate, the QA representative clarifies the process for the employee to assure understanding. If the situation appears to be a data integrity concern, the QA representative initiates a Data Integrity Investigation following the procedures specified in SOP\_Data\_Integrity\_Investigations\_20091009.doc.

**Data Integrity Investigations.** Follow-up investigations are conducted for all reported instances of ethical concern related to data integrity. Investigations are performed in a confidential manner by senior management according to a documented procedure. The outcome of the investigation is documented and reported to the company president who has the ultimate responsibility for determining the final course of action in the matter. Investigation documentation includes corrective action records, client notification information and disciplinary action outcomes, which is archived for a period of five years.

The investigations are conducted by the senior staff and supervisory personnel from the affected area. The investigations team includes the Laboratory Director and the Quality Assurance Director. Investigations are conducted in a confidential manner until it is completed and resolved.

The investigation includes a review of the primary information in question by the investigations team. The team performs a review of associated data and similar historical data to determine if patterns exist. Interviews are conducted with key staff to determine the reasons for the observed practices.

Following data compilation, the investigations team reviews all information to formulate a consensus conclusion. The investigation results are documented along with the recommended course of action.

**Corrective Action, Client Notification & Discipline.** Investigations that reveal systematic data integrity issues will be referred for an Incident Report. If the investigation indicates that an impact to data has occurred and the defective data has been released to clients, the client will be notified by the Laboratory Director.

In all cases of data integrity violations, some level of disciplinary action will be conducted on the responsible individual. The level of discipline will be consistent with the violation and may range from retraining and/or verbal reprimand to termination. A zero tolerance policy is in effect for unethical actions.

## 4.0 JOB DESCRIPTIONS OF KEY STAFF

4.1 ***Requirement:*** Descriptions of key positions within the organization are defined to ensure that clients and staff understand duties and the responsibilities of the management staff and the reporting relationships between positions.

**President/Chief Executive Officer.** Responsible for all laboratory operations and business activities. Establishes the company mission and objectives in response to business needs. Direct supervision of the Vice President of Operations, each laboratory director, client services, management information systems, quality assurance and health and safety.

**Vice President, Operations (Corporate).** Reports to the company president. Establishes laboratory operations strategy. Operational responsibility for Orlando, Florida, Marlborough, Massachusetts, Santa Clara, California and Houston, Texas laboratories. Assumes the responsibilities of the CEO in his absence.

**Laboratory/Technical Director (Local).** Reports to the Vice President of Operations. Establishes laboratory operations strategy. Direct supervision of organic chemistry, inorganic chemistry, and sample management. Minimum Qualifications: BS in Chemistry or other physical science and 10 years experience in Environmental Chemistry.

**Vice President, Chief Information Officer (Corporate).** Reports to the company president. Develops the IT software and hardware agenda. Provides system strategies to compliment company objectives. Maintains all software and hardware used for data handling.

**Director, Quality Assurance (Corporate).** Reports to the company president. Establishes the company quality agenda, develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities, monitors the quality system, provides quality system feedback to management to be used for process improvement and oversees health and safety. Assumes the responsibilities of the CEO in the absence of the CEO and the Vice President Operations.

### **Laboratory Manager**

Manages the day to day operations of the technical staff and client services, for a laboratory engaged in the analysis of soil, ground water, surface water, and waste water samples for commercial, industrial, and municipal clients in support of a wide variety of projects from various state and Department of Defense sites, Ensures that reported data meets client and regulatory requirements, Acts as technical resource for sales staff, clients, and data validators, Directs development of new methods and continual improvement of existing processes

**Manager, Volatile Organics .** Reports to the laboratory director. Directs the operations of the volatile organics group, consisting of organics preparation and instrumental analysis. Establishes daily work schedule. Supervises method implementation, application, and data production. Responsible for following Quality System requirements. Maintains laboratory

instrumentation in an operable condition. Minimum Qualifications: BS in Chemistry or other physical science and 5 years experience in Environmental Chemistry.

**Manager, Semi-Volatile Organics** . Reports to the laboratory director. Directs the operations of the Semi-volatile organics analysis and Organic extractions group. Establishes daily work schedule. Supervises method implementation, application, and data production. Responsible for following Quality System requirements. Maintains laboratory instrumentation in an operable condition. Minimum Qualifications: BS in Chemistry or other physical science and 5 years experience in Environmental Chemistry.

**Inorganics Supervisor**. Reports to the laboratory director. Supervises the operations of the inorganics group, consisting of wet chemistry and the metals laboratories. Maintains laboratory instrumentation in an operable condition. Establishes daily analysis schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples using valid, documented methodology. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality System requirements. Minimum Qualifications: BS in Chemistry or other physical science and 5 years experience in Environmental Chemistry.

**Manager, Sample Management**. Reports to the laboratory director. Develops, maintains and executes all procedures required for receipt of samples, verification of preservation, and chain of custody documentation. Responsible for maintaining and documenting storage, and courier services. Minimum Qualifications: BS degree and 5 years experience in Environmental Chemistry.

**Principal Analysts**. Reports to the department Supervisors. Maintains laboratory instrumentation in an operable condition, analyze samples on a daily basis. Implement methods, applications, and data production. Analyze samples using valid, documented methodology. Mentor junior analysts and reviews data for compliance to quality and methodological requirements. Responsible for following Quality System requirements. Minimum Qualifications: BS in Chemistry or other physical science and 1 years experience in Environmental Chemistry.

**Health & Safety Officer**. Reports to the Director of Quality Assurance. Responsible for developing company safety program and chemical hygiene plan. Reviews and updates these plans annually. Responsible for employee training on relevant health and safety topics. Documents employee training. Manages laboratory waste management program.

**Quality Assurance Officer (Local)**. Reports to the Director of Quality Assurance. Performs quality control data review for trend monitoring purposes. Conducts internal audits and prepares reports for management review. Oversees proficiency testing program. Process quality control data for statistical purposes.

#### 4.2 **Employee Screening, Orientation, and Training.**

All potential laboratory employees are screened and interviewed by human resources and technical staff prior to their hire. The pre-screen process includes a review of their qualifications including education, training and work experience to verify that they have adequate skills to perform the tasks of the job.

Newly hired employees receive orientation training beginning the first day of employment by the Company. Orientation training consists of initial health and safety training including general laboratory safety, personal protection and building evacuation. Orientation also includes quality assurance program training, data integrity training, and an overview of the Company's goals, objectives, mission, and vision.

All technical staff receives training to develop and demonstrate proficiency for the methods they perform. New analysts work under supervision until the supervisory staff is satisfied that a thorough understanding of the method is apparent and method proficiency has been demonstrated, through a precision and accuracy study that has been documented, reviewed and approved by the QA Officer. Data from the study is compared to method acceptance limits. If the data is unacceptable, additional training is required. The analyst may also demonstrate proficiency by producing acceptable data through the analysis of an independently prepared proficiency sample.

Individual proficiency is demonstrated annually for each method performed. ODOC Data from initial and continuing proficiency demonstrations are archived in the individual's training folder.

#### 4.3 **Training Documentation.** The human resources department prepares a training file for every new employee. This administrative information related to qualifications, experience, external training courses, and education are placed into the file. Verification documentation for orientation, health & safety, quality assurance, and ethics training is also included in the file.

Additional technical training documentation is filed locally. This includes documentation of SOP understanding, data for initial and continuing demonstrations of proficiency, performance evaluation study data and notes and attendance lists from group training sessions.

The Quality Assurance Department maintains the employee training directory. This directory is a comprehensive inventory of training documentation for each individual employee. These files enable the supervisors to obtain current status information on technical training data for individual employees.

## 5.0 SIGNATORY APPROVALS

**Requirement:** Procedures have been developed for establishing the traceability of data and documents. The procedure consists of a signature hierarchy, indicating levels of authorization for signature approvals of data and information within the organization. Signature authority is granted for approval of specific actions based on positional hierarchy within the organization and knowledge of the operation that requires signature approval. A log of signatures and initials of all employees is maintained by the HR Staff for cross-referencing purposes.

### 5.1 Signature Hierarchy.

**President/Chief Executive Officer.** Authorization for contracts and binding agreements with outside parties. Approval of final reports, quality assurance policy, SOPs, project specific QAPs, data review and approval in lieu of technical managers. Note: Contract signature authority resides with Company officers only, which include the President/CEO, Chief Financial Officer and Vice President Administration.

**Vice President, Operations/Laboratory Director.** Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Establishes and implements technical policy.

**Director, Quality Assurance.** Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers.

**Director, Client Services.** QAP and sampling and analysis plan approval. Project specific contracts, pricing, and price modification agreements. Approval and acceptance of incoming work, Client services policy.

**Managers, Technical Departments.** Methodology and department specific QAPs. Data review and approval, department specific supplies purchase. Technical approval of SOPs.

**Manager, Sample Management.** Initiation of laboratory sample custody and acceptance of all samples. Approval of department policies and procedures. Department specific supplies purchase.

**Manager, Health & Safety.** Approval of health and safety policy in the absence of the President and QA Director. Approval of health and safety SOPs. Waste manifesting and approval.

**Supervisors, Technical Departments.** Data review approval, purchasing of expendable supplies.

5.2 **Signature Requirements.** All laboratory activities related to sample custody and generation or release of data must be approved using either initials, signatures or electronic, password protected procedures. The individual, who applies his signature initial or password to an activity or document, is authorized to do so within the limits assigned to them by their supervisor. All written signatures and initials must be applied in a readable format that can be cross-referenced to the signatures and initials log if necessary.

5.3 **Signature and Initials Log.** The Laboratory maintains a signature and initials log. New employee signatures and initials are appended to the log on the first day of employment. Signature of individuals no longer employed by the company are retained, but annotated with their date of termination.

## 6.0 DOCUMENTATION & DOCUMENT CONTROL

**Requirement.** Document control policies have been established which specify that any document used as an information source or for recording analytical or quality control information must be managed using defined document control procedures. Accordingly, policies and procedures required for the control, protection, and storage of any information related to the production of analytical data and the operation of the quality system to assure its integrity and traceability have been established and implemented in the laboratory. The system contains sufficient controls for managing, archiving and reconstructing all process steps which contributed to the generation of an analytical test result. Using this system, an audit trail for reported data can be produced, establishing complete traceability for the result.

6.1 **Administrative Records.** Administrative (non-analytical) records are managed by the quality assurance department. These records consist of electronic documents which are retained in a limited access electronic directory or paper documents, which are released to the technical staff upon specific request.

**Form Generation, Modification & Control.** The quality assurance group approves and manages all forms used as either stand-alone documents or in logbooks to ensure their traceability. Forms are generated as computer files only and are maintained in a limited access master directory. The QA staff also manages and approves modifications to existing forms. Obsolete editions of modified forms are retained for seven years.

New forms must include the name Accutest Laboratories and appropriate spaces for signatures of approval and dates. Further design specifications are the responsibility of the originating department.

The technical staff is required to complete all forms to the maximum extent possible. If information for a specific item is unavailable, the analyst is required to "Z" the information block. The staff is also required to "Z" the uncompleted portions of a logbook or logbook form if the day's analysis does not fill the entire page of the form.

**Logbook Control.** All laboratory logbooks are controlled documents that are comprised of approved forms used to document specific processes. New logs are numbered and issued to a specific individual who is assigned responsibility for the log. Old logs are returned to QA for entry into the document archive system where they are retained for seven (7) years. Laboratory staff may hold a maximum of two consecutively dated logbooks of the same type in the laboratory including the most recently issued book to simplify review of recently completed analysis.

**Controlled Documents.** Key laboratory documents that are distributed internally and externally are numbered and dated for tracking purposes. Individuals receiving documents, who must be assured they have the most recent, receive a file server location where the most current revisions reside. Control is maintained through a document - revision numbering and dating procedure. Key documents are also distributed as uncontrolled documents if the recipient does not require updated copies when changes occur.

**Quality Systems Manual (QSM).** All QSMs are titled, revision indicated, and dated prior to distribution. Electronic versions are distributed as read only files that are password protected. **The online version is always the most current.**

**Standard Operating Procedures (SOPs).** SOPs are maintained by title, revision, and date. One copy of the SOP is placed into each the department. SOPs are reviewed annually.

The original, signed copy of the SOP is maintained in the master SOP binder by the QA staff. The QA staff collects outdated versions of SOPs as they are replaced and archived for a period of seven (7) years in the QA archives. Electronic versions of outdated SOPs are moved from the active SOP directory to the inactive directory identified by year.

- 6.2 Technical Records.** All records related to the analysis of samples and the production of an analytical result are archived in secure document storage or on electronic media and contain sufficient detail to produce an audit trail which re-creates the analytical result. These records include information related to the original client request, bottle order, sample login and custody, storage, sample preparation, analysis, data review and data reporting.

Each department involved in this process maintains controlled documents which enable them to maintain records of critical information relevant to their department's process.

- 6.3 Quality Control Support Data & Records.** All information and data related to the quality system is stored in a restricted access directory on the network server. Information on this directory is backed-up daily. Users of the quality assurance information and data have "read-only" access to the files contained in the directory. The QA staff and the laboratory director have write capability in this directory.

This directory contains all current and archived quality system manuals, SOPs, control limits, MDL studies, precision and accuracy data, official forms, internal audit reports, proficiency test scores and metrics calibration information.

- 6.4 *Analytical Records.*** All data related to the analysis of field samples are retained as either paper or electronic records that can be retrieved to compile a traceable audit trail for any reported result. All information is linked to the client job and sample number, which serves as a reference for all sample related information tracking.

Critical times in the life of the sample from collection through analysis to disposal are documented. This includes date and time of collection, receipt by the laboratory, preparation times and dates, analysis times and dates and data reporting information. Analysis times are calculated in hours for methods where holding time is specified in hours ( $\leq 72$  hours).

Sample preparation information is recorded in a separate controlled logbook. It includes sample identification numbers, types of analysis, preparation and cleanup methods, sample weights and volumes, reagent lot numbers and volumes and any other information pertinent to the preparation procedure.

Information related to the identification of the instrument used for analysis is permanently attached to the electronic record. The record includes an electronic data file that indicates all instrument conditions employed for the analysis, including the type of analysis conducted. The analyst's identification is electronically attached to the record. The instrument tuning and calibration data is electronically linked to the sample or linked through paper logs which were used in the documentation of the analysis. Quality control and performance criteria are permanently linked to the paper archive or electronic file.

Paper records for the identity, receipt, preparation and evaluation of all standards and reagents used in the analysis are documented in prepared records and maintained in controlled documents or files. Lot number information linking these materials to the analysis performed is recorded in the logbooks associated with the samples in which they were used.

Manual calculations or peak integrations that were performed during the data review are retained as paper or scanned documents and included as part of the electronic archive. Signatures for data review are retained on paper or as scanned versions of the paper record for the permanent electronic file.

- 6.5 **Confidential Business Information (CBI).** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as secured Adobe Acrobat .PDF format only.

- 6.6 **Software Change Documentation & Control.** Changes to software are documented as text within the code of the program undergoing change. Documentation includes a description of the change, reason for change and the date the change was placed into effect. Documentation indicating the adequacy of the change is prepared following the evaluation by the user who requested the change.

- 6.7 **Report and Data Archiving.** Accutest Laboratories produces digital files of all raw and processed data which is maintained for a minimum period of seven (7) years. The archived files consist of all raw data files and source documents associated with the analysis of field samples and proficiency test samples. Data files and source documents associated with method calibration and project and method quality control are also archived. After seven years, the files are discarded unless contractual arrangements exist which dictate different requirements. Client or regulatory agency specific data retention practices are employed for several government organizations such as the Department of Defense and the Massachusetts Department of Environmental Protection that require a retention period of ten (10) years. Data archiving may also be extended up to ten (10) years for specific commercial clients in response to contractual requirements.

Complete date and time stamped PDF reports are generated from the laboratory information management system (LIMS) using the source documents archived on the document server. These source documents are maintained on a document server and archived to primary and clone tapes. The primary tapes remain on premises while the clone tapes are taken to a secure offsite location for permanent storage. Both the primary and clone tapes remain in storage for the remainder of the archive period.

- 6.8 **Training.** The company maintains a training record for all employees that documents that they have received instruction on administrative and technical tasks that are required for the job they perform. Training records for individuals employed by the company are retained for a period of six months following their termination of employment.

**Training File Origination.** The Human Resources Group (HR) initiates training files. The QA staff, through the Assistant Quality Assurance officer, retains the responsibility for the maintenance and tracking of all training related documentation in the file. The file is begun on the first day of employment. Information required for the file includes a copy of the individual's most current resume, detailing work experience and a copy of any college diplomas and transcript(s). Information added on the first day includes documentation of health and safety training, quality assurance training and a signed data integrity training and ethical conduct agreement.

Training documentation, training requirements, analyst proficiency information and other training related support documentation is archived locally.

- 6.9 **Technical Training.** The supervisor of each new employee is responsible for developing a training plan for each new employee. The supervisor evaluates the employees training progress at regular frequencies. Supporting documentation, including demonstration of capability and precision and accuracy studies, which demonstrate an analyst's proficiency for a specific test, are added to the training file as completed. Employees and supervisors verify documentation of understanding for all assigned standard operating procedure. Certificates or diplomas for any off-site training are also added to the file.

## 7.0 REFERENCE STANDARD TRACEABILITY

**Requirement:** Documented procedures, which establish traceability between any measured value and a national reference standard, are established by the laboratory as required. All metric measurements are traceable to NIST reference weights or thermometers that are calibrated on a regular schedule. All chemicals used for calibration of a quantitative process are traceable to an NIST reference that is documented by the vendor using a certificate of traceability. The laboratory maintains a documentation system that establishes the traceability links. The procedures for verifying and documenting traceability are documented in standard operating procedures.

7.1 **Traceability of Metric Measurements - Thermometers.** ANC uses NIST thermometers to calibrate commercially purchased thermometers prior to their use in the laboratory. If necessary, thermometers are assigned correction factors that are determined during their calibration using an NIST thermometer as the standard. The correction factor is documented on a tag attached to the thermometer. The correction factor is applied to temperature measurements before recording the measurement in the temperature log. Calibration of each thermometer is verified and documented on a regular schedule. The NIST thermometer is checked for accuracy by a qualified vendor every five (5) years following the specifications for NIST thermometer calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005.

7.2 **Traceability of Metric Measurements – Calibration Weights.** Accutest uses calibrated weights, which are traceable to NIST standard weights to calibrate all balances used in the laboratory. Balances are calibrated to specific tolerances within the intended use range of the balance. Calibration checks are required on each day of use. If the tolerance criteria are not achieved, corrective action specified in the balance calibration SOP is applied before the balance can be used for laboratory measurements. Recalibration of all calibration weights is conducted and documented on a biannual basis.

7.3 **Traceability of Chemical Standards.** All chemicals, with the exception of bulk dry chemicals and acids, purchased as reference standards for use in method calibration must establish traceability to NIST referenced material through a traceability certificate. Process links are established that enable a calibration standard solution to be traced to its NIST reference certificate.

Chemical standards used for analysis must meet the purity specifications of the method. These specifications must be stated in the reagents section of the method SOP.

7.4 **Assignment of Reagent and Standard Expiration Dates.** Expiration date information for all purchased standards, prepared standard solutions and selected reagents is provided to Accutest by the vendor as a condition of purchase. Neat materials and inorganic reagents are not required to be purchased with expiration dates. Prepared solutions are labeled with the expiration date provided by the manufacturer. In-house prepared solutions are assigned

expiration dates that are consistent with the method that employs their use unless documented experience indicates that an alternate date can be applied. If alternate expiration dates are employed, their use is documented in the method SOP. Expiration dates for prepared inorganic reagents, which have not exhibited instability, are established at two years from the date of preparation for tracking purposes.

The earliest expiration date has been established as the limiting date for assigning expiration dates to prepared solutions. The assignments of expiration dates that are later than the expiration date of any derivative solution or material are prohibited.

**7.5** *Documentation of Traceability*. Traceability information is documented in individual logbooks designated for specific measurement processes. The quality assurance group maintains calibration documentation for metric references in separate logbooks.

Balance calibration verification is documented in logbooks that are assigned to each balance. The individual conducting the calibration is required to initial and date all calibration activities. Any defects that occur during calibration are also documented along with the corrective action applied and a demonstration of return to control. Annual service reports and certificates are retained on the file server by the QA staff.

Temperature control is documented in logbooks assigned to the equipment being monitored. A calibrated thermometer is assigned to each individual item. Record date and initials of the individual conducting the measurement on a daily or as used basis. Corrective action, if required, is also documented including the demonstration of return to control.

Initial traceability of chemical standards is documented via a vendor-supplied certificate (not available for bulk dry chemicals and acids) that includes lot number, expiration date and certified concentration information. Solutions prepared using the vendor supplied chemical standards are documented in logbooks assigned to specific analytical processes. The documentation includes links to the vendor's lot number, an internal lot number, dates of preparation, expiration date, and the preparer's initials.

Accutest employs commercially prepared standard solutions whose traceability can be demonstrated through a vendor supplied certificate of analysis that includes an experimental verification of the standard's true concentration. The test value for the verification analysis must agree within 1% of the vendor's true value before it can be employed for calibration purposes. If the test value differs from the nominal value by more than 1%, then the test value is used as the true value in laboratory calibrations and calculations. Purchased standards which do not have a certificate of analysis cannot be used for calibration or calibration verification purposes and are rejected or returned to the vendor. Supervisors conduct regular reviews of logbooks, which are verified using a signature and date.

## 8.0 TEST PROCEDURES, METHOD REFERENCES, AND REGULATORY PROGRAMS

**Requirements.** The laboratory employs client specified or regulatory agency approved methods for the analysis of environmental samples. A list of active methods is maintained, which specifies the type of analyses performed and cross-references the methods to applicable environmental regulations. Routine procedures used by the laboratory for the execution of a method are documented in standard operating procedures. Method performance and sensitivity are demonstrated annually where required. Defined procedures for the use of method sensitivity limits for data reporting purposes are established by the Director of Quality Assurance and used consistently for all data reporting purposes.

- 8.1 **Method Selection & Application.** Accutest employs methods for environmental sample analysis that are consistent with the client's application, which are appropriate and applicable to the project objectives. Accutest informs the client if the method proposed is inappropriate or outdated and suggests alternative approaches.

Accutest employs documented, validated regulatory methods in the absence of a client specification and informs the client of the method selected. These methods are available to the client and other parties as determined by the client. Documented and validated in-house methods may be applied if they are appropriate to the project. The client is informed of the method selection.

- 8.2 **Standard Operating Procedures.** Standard operating procedures (SOP) are prepared for routine methods executed by the laboratory, processes related to laboratory operations and sample or data handling. All SOPs are formatted to meet the specifications established by the National Environmental Laboratory Accreditation Conference, which are detailed in Chapter Five – Quality Systems of the established Standards. The procedures describe the process steps in sufficient detail to enable an individual, who is unfamiliar with the procedure to execute it successfully.

SOPs are evaluated annually and edited if necessary. Reviewed SOPs that do not require modification include an evaluation summary form indicating that an evaluation was conducted and modifications were not needed. SOPs can be edited on a more frequent basis if changes are required for any reason. These may include a change to the methodology, elimination of systematic errors that dictate a need for process changes or modifications to incorporate a new version of the method promulgated by the originating regulatory agency. Procedural modifications are indicated using a revision number. SOPs are available for client review at the Accutest facility upon request.

**8.3 Method Validation.** Standard methods from regulatory sources are primarily used for all analysis. Standard methods do not require validation by the laboratory. Non-standard, in-house methods are validated prior to use. Validation is also performed for standard methods applied outside their intended scope of use. Validation is dependent upon the method application and may include analysis of quality control samples to develop precision and accuracy information for the intended use. A final method validation report is generated, which includes all data in the validation study. A statement of adequacy and/or equivalency is included in the report. A copy of the report is archived in the quality assurance directory of the company server.

Non-standard methods are validated prior to use. This includes the validation of modified standard methods to demonstrate comparability with existing methods. Demonstrations and validations are performed and documented prior to incorporating technological enhancements and non standard methods into existing laboratory methods used for general applications. The demonstration includes method specific requirements for assuring that significant performance differences do not occur when the enhancement is incorporated into the method. Validation is dependent upon method application and may include the analysis of quality control samples to develop precision and accuracy information for intended use.

The study procedures and specifications for demonstrating validation include comparable method sensitivity, calibration response, method precision; method accuracy and field sample consistency for several classes of analytical methods are detailed in this document. These procedures and specifications may vary depending upon the method and the modification.

**8.4 Estimated Uncertainty.** A statement of the estimated uncertainty of an analytical measurement accompanies the test result when required. Estimated uncertainty is derived from the performance limits established for spiked samples of similar matrices. The degree of uncertainty is derived from the negative or positive bias for spiked samples accompanying a specific parameter. When the uncertainty estimate is applied to a measured value, the possible quantitative range for that specific parameter at that measured concentration is defined. Well recognized regulatory methods that specify values for the major sources of uncertainty and specify the data reporting format do not require a further estimate of uncertainty.

**8.5 Demonstration of Capability.** Confirmation testing is conducted to demonstrate that the laboratory is capable of performing the method before its application to the analysis of environmental samples. The results of the demonstration tests are compared to the quality control specifications of the method to determine if the performance is acceptable.

Capability demonstrations are conducted initially for each method on every instrument and annually on a method specific basis thereafter. Acceptable demonstrations are documented for individual training files and retained by the QA staff. New analytes, which are added to the list of analytes for an accredited method, are evaluated for applicability through a demonstration of capability similar to those performed for accredited analytes.

- 8.6 **Method Detection Limit Determination.** Annual method detection limit (MDL) studies are performed as appropriate for routine methods used in the laboratory. MDL studies are also performed when there is a change to the method that affects how the method is performed or when an instrumentation change that impacts sensitivity occurs. The procedure used for determining MDLs is described in 40 CFR, Part 136, Appendix B. Studies are performed for each method on water, soil and air matrices for every instrument that is used to perform the method. MDLs are established at the instrument level. The highest MDL of the pooled instrument data is used to establish a laboratory MDL. The quality assurance staff manages the annual MDL determination process and is responsible for retaining MDL data on file. Approved MDLs are appended to the LIMS and used for data reporting purposes.
- 8.7 **Instrument Detection Limit Determination.** Instrument detection limits (IDLs) are determined for all inductively coupled argon plasma emission spectrophotometers and mass spectrometers. The IDL is determined for the wavelength (emission) of each element and the ion (mass spectrometry) of each element used for sample analysis. The IDL data is used to estimate instrument sensitivity in the absence of the sample matrix. IDL determinations are conducted at the frequency specified in the appropriate SOPs' for ICP analysis.
- 8.8 **Method Reporting Limit.** The method reporting limit for organic methods is determined by the concentration of the lowest calibration standard in the calibration curve. This value is adjusted based on several sample preparation factors including sample volume, digestion, distillation or dilution. The low calibration standard is selected by department managers as the lowest concentration standard that can be used for calibration while continuing to meet the calibration linearity criteria of the method being used. The validity of the method reporting limits are confirmed through the analysis of a spiked quality control sample at the method reporting limit concentration. By definition, detected analytes at concentrations below the low calibration standard cannot be accurately quantitated and are qualified as estimated values.
- The reporting limits for inorganic methods is defined as the concentration which is greater than or equal to the MDL where method quality control criteria has been achieved. The reporting limit for general chemistry methods employing multiple point calibrations must be greater than or equal to the concentration of the lowest standard of the calibration range.
- 8.9 **Reporting of Quantitative Data.** Analytical data for all methods is reported without qualification to the reporting limit established for each method. Data, for organic methods may be reported to the established method detection limit depending upon the client's requirements provided that all qualitative identification criteria for the detected parameter have been satisfied. All parameters reported at concentrations between the reporting limit and the method detection limit is qualified as estimated.

Data for inorganic methods are reported to the established method reporting limits. Inorganic data for specific methods may also be reported to the established method detection limit at client request. However, this data is always qualified as estimated.

Measured concentrations of detected analytes that exceed the upper limit of the calibration range are either diluted into the range and reanalyzed or qualified as an estimated value. The only exception to this applies to ICP analysis, which can be reported to the upper limit of the experimentally determined linear range without qualification.

**8.10 Precision and Accuracy Studies.** Annual precision and accuracy (P&A) studies, which demonstrate the laboratories ability to generate acceptable data, are performed for all routine methods used in the laboratory. The procedure used for generating organic P&A data is referenced in the majority of the regulatory methodology in use. The procedure requires quadruplicate analysis of a sample spiked with target analytes at a concentration in the working range of the method. This data may be compiled from a series of existing blank spikes or laboratory control samples. Accuracy (percent recovery) of the replicate analysis is averaged and compared to established method performance limits. Values within method limits indicate an acceptable performance demonstration. Precision and accuracy data is also used to annually demonstrate analytical capability for individual analysts. Annual demonstration of capability data is archived in individual training files.

**8.11 Method Sources & References.** The Quality Assurance Staff maintains a list of active methods used for the analysis of samples. This list includes valid method references from sources such as USEPA, ASTM or Standard Methods designations and the current version and version date.

Updated versions of approved reference methodology are placed into use as changes occur. The Quality Assurance Director informs operations management of changes in method versions as they occur. The operations management staff selects an implementation date. The operations staff is responsible for completing all method use requirements prior to the implementation date. This includes modification of SOPs, completion of MDL and precision and accuracy studies and staff training. Documentation of these activities is provided to the QA staff who retains this information on file. The updated method is placed into service on the implementation date and the old version is de-activated.

Multiple versions of selected methods may remain in use to satisfy client specific needs. In these situations, the default method version becomes the most recent version. Client specific needs are communicated to the laboratory staff using method specific analytical method codes, which clearly depict the version to be used. The old method version is maintained as an active method until the specified client no longer requires the use of the older version.

Accutest will not use methodology that represents significant departures from the reference method unless specifically directed by the client. If clients direct the laboratory to use a method modification that represents a significant departure from the reference method, the request will be documented in the client file.

**8.12 Analytical Capabilities.** Appendix III provides a detailed listing of the methodology employed for the analysis of test samples.

## 9.0 SAMPLING, SAMPLE MANAGEMENT, LOGIN, CUSTODY, STORAGE AND DISPOSAL

**Requirement.** The laboratory must employ a system which ensures that client samples are adequately evaluated, acknowledged, and secured upon delivery to the laboratory. The system also assures that product chain of custody is maintained and that sample receipt conditions and preservation status are documented and communicated to the client and internal staff. The login procedure assigns, documents, and maps the specifications for the analysis of each unique sample to assure that the requested analysis is performed on the correct sample and enables the sample to be tracked throughout the laboratory analytical cycle. The system includes procedures for reconciling defects in sample condition or client provided data, which are identified at sample arrival. The system specifies the procedures for proper sample storage, transfer to the laboratory, and disposal after analysis. The system is also documented in standard operating procedures.

- 9.1 **Order Receipt and Entry.** New orders are initiated and processed by the client services group. The new order procedure includes mechanisms for providing bottles to clients, which meet the size, cleanliness, and preservation specifications for the analysis to be performed.

For new orders, the project manager prepares a bottle request form, which is submitted to sample management. This form provides critical project details to the sample management staff, which are used to prepare and assemble the sample bottles for shipment to the client prior to sampling.

The bottle order is assembled using bottles that meet USEPA specifications for contaminant free sample containers. Accutest-Northern California uses commercially supplied pre-cleaned bottles.

Reagent water for trip and field blanks is poured into appropriately labeled containers. Bottles may be packed into ice chests with blank chain of custody forms and the original bottle order form or bottle shipped alone. Completed bottle orders are delivered to clients using Accutest couriers or commercial carriers for use in field sample collection.

- 9.2 **Sampling.** Accutest-Northern California does not have a sampling staff.

- 9.3 **Sample Receipt and Custody.** Samples are delivered to the laboratory using a variety of mechanisms including Accutest couriers, commercial shippers, and client self-delivery. Documented procedures are followed for arriving samples to assure that custody and integrity are maintained and handling/ preservation requirements are documented and maintained.

Sample custody documentation is initiated when the individual collecting the sample collects field samples. Custody documentation includes all information necessary to provide an unambiguous record of sample collection, sample identification, and sample collection

chronology. Initial custody documentation employs either Accutest or client generated custody forms.

Accutest generates a chain of custody in situations where the individuals who collected the sample did not generate custody documentation in the field.

Accutest defines sample custody as follows:

- ∴ The sample is in the actual custody or possession of the assigned responsible person,
- ∴ The sample is in a secure area.

The Accutest facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. The sample management team is located in the vicinity of all sample storage during work hours. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors must be accompanied by their hosts at all times. After hours, building access is controlled using a computerized alarm system. This system limits building access to individuals with a pre-assigned authorization status. After hours visitors are not authorized to be in the building. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Upon arrival at Accutest, the sample custodian reviews the chain of custody for the samples received to verify that the information on the form corresponds with the samples delivered. This includes verification that all listed samples are present and properly labeled, checks to verify that samples were transported and received at the required temperature, verification that the sample was received in proper containers, verification that sufficient volume is available to conduct the requested analysis, and a check of individual sample containers to verify test specific preservation requirements including the absence of headspace for volatile compound analysis.

Sample conditions and other observations are documented on the chain of custody by the sample custodian prior to completing acceptance of custody and in the LIMS. The sample custodian accepts sample custody upon verification that the custody document is correct. Discrepancies or non-compliant situations are documented and communicated to the Accutest project manager, who contacts the client for resolution. The resolution is documented in the report archive directory on the file server and communicated to sample management for execution.

During initial login, each sample is assigned a unique number and is labeled with that number.

- 9.4 ***Laboratory Preservation of Improperly Preserved Field Samples.*** Accutest will attempt to preserve field samples that were received without proper preservation to the extent that it is feasible and supported by the methods in use. Laboratory preservation of improperly preserved or handled field samples is routinely performed for metals samples. Special handling procedures may also be applied to improperly preserved volatile organics.

Aqueous metals samples that were not nitric acid preserved to pH 2 in the field are laboratory preserved and held for twenty-four (24) hours to equilibrate prior to analysis (per Federal Register). Aqueous metals samples requiring field filtration may be filtered in the laboratory within seventy-two (72) hours of receipt provided that the sample has not been acid preserved.

Unpreserved volatile organics samples may be analyzed within seven (7) days to minimize degradation of volatile organics if the laboratory is notified in advance of the failure to preserve upon collection. Laboratory preservation of unpreserved aqueous samples is not possible. A pH check of volatile organic samples prior to analysis will compromise the sample by allowing volatile organics to escape during the check. If the laboratory is not notified of the failure to field preserve an aqueous volatile organic sample, the defect will not be identified until sample analysis has been completed and the data is qualified accordingly.

- 9.5 ***Sample Tracking Via Status Change.*** An automated, electronic LIMS procedure records sample exchange transactions between departments and changes in analytical status. This system tracks all preparation, analytical, and data reporting procedures to which a sample is subjected while in the possession of the laboratory.

Sample tracking is initiated at login where all chronological information related to sample collection dates and holding times are entered into the LIMS. This information is entered on an individual sample basis.

- 9.6 ***Sample Acceptance Policy.*** Incoming samples must satisfy ANC's sample acceptance criteria before being logged into the system. Sample acceptance is based on the premise that clients have exercised proper protocols for sample collection. This includes complete documentation, sufficient volume, proper chemical preservation, temperature preservation, sample container sealing and labeling, and appropriate shipping container packing.

The sample management staff will make every attempt to preserve improperly preserved samples upon arrival. However, if preservation is not possible, the samples may be refused unless the client authorizes analysis. No samples will be accepted if holding times have been exceeded or will be exceeded before analysis can take place unless the client authorizes analysis.

Sample acceptance criteria include proper custody and sample labeling documentation. Proper custody documentation includes an entry for all physical samples delivered to the laboratory with an identification code that matches the sample bottle and a date and signature of the individual who collected the sample and delivered them to the laboratory.

**Accutest-Northern California reserves the right to refuse any sample which in its sole and absolute discretion and judgment is hazardous, toxic and poses or may pose a health, safety or environmental risk during handling or processing. The company will not accept samples for analysis using methodology that is not performed by the laboratory or for methods that lab does not hold valid accreditations unless arrangements have been made to have the analysis conducted by a qualified subcontractor.**

- 9.7 ***Assignment of Unique Sample Identification Codes.*** Unique identification codes are assigned to each sample to assure traceability and unambiguously identify the tests to be performed in the laboratory.

The sample identification coding process begins with the assignment of a unique alphanumeric job number. A job is defined as a group of samples received on the same day, from a specific client pertaining to a specific project. A job may consist of groups of samples sampled over a multi-day period. The first character of the job number is an alpha-character that identifies the laboratory facility. Accutest Northern California has job numbers that start with “C”. The next characters are numeric and sequence by one number with each new job.

Unique sample numbers are assigned to each bottle collected as a discrete entity from a designated sample point. This number begins with the job number and incorporates a second series of numbers beginning at one and continuing chronologically for each point of collection.

Alpha suffixes may be added to the sample number to identify special designations such as subcontracted tests, in-house QC checks, or re-logs.

- 9.8 ***Subcontracted Analysis.*** Subcontract laboratories are employed to perform analysis not performed by Accutest. The quality assurance staff evaluates subcontract laboratories via CDPH-ELP and NELAP certification to assure their quality processes meet the standards of the environmental laboratory industry prior to engagement. Throughout the subcontract process, Accutest follows established procedures to assure that sample custody is maintained and the data produced by the subcontractor meets established quality criteria.

*Subcontracting Procedure.* Subcontracting procedures are initiated through several mechanisms, which originate with sample management. Samples for analysis by a subcontractor are logged into the Accutest system using regular login procedures. If subcontract parameters are part of the project or sample management has received subcontracting instructions for a specific project, a copy of the chain of custody is given to the appropriate project manager with the subcontracted parameters highlighted. This procedure triggers the subcontract process at the project management level. The project manager contacts an approved subcontractor that carries accreditation in the venue of the project location to place the subcontract order. A subcontract chain of custody is simultaneously prepared in electronic format and filed with the original chain of custody. The subcontract chain of custody is forwarded along with the samples.

Sample management signs the subcontract chain of custody and ships the sample(s) to the subcontractor. The subcontract CoC is filed with the original CoC and the request for subcontract.

Clients are verbally notified of the need to subcontract analysis as soon as the need is identified by the client services staff. This may occur during the initial project setup or at the time of login if the project setup had not been initiated through the client services staff. Copies of the subcontract CoC and the original CoC, which are electronically distributed to clients, this constitutes documented client notification of the laboratories' intent to subcontract analyses.

Subcontractor data packages are reviewed by the Reporting Staff to assess completeness. If completeness defects are detected, the subcontractor is asked to immediately upgrade the data package.

Subcontract data is wholly incorporated into the final report by Adobe PDF Acrobat.

*Subcontract Laboratory Evaluation.* The subcontract laboratory must provide ANC with proof of a valid certification to perform the requested analysis for the venue where they were collected, and a copy of the laboratory's Quality Systems Manual. If possible, the QA staff may conduct a site visit to the laboratory to inspect the quality system. Qualification of a subcontract laboratory is bypassed if the primary client directs Accutest-Northern California to employ a specific subcontractor. All subcontracted items for testing covered under DoD will only be submitted to a DoD-ELAP laboratories.

- 9.9** **Sample Storage.** Following sample transfer to the sample custodian, samples are assigned to various refrigerated storage areas depending upon the test to be performed and the matrix of the samples. Samples remain in storage until the laboratory technician removes them for analysis.

The Accutest facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors must be accompanied by their hosts at all times. After hours, building access is controlled using an alarm system. After hours visitors are not authorized to be in the building. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Samples for volatile organics analysis are placed in specifically designated refrigerators. These samples are segregated according to matrix to limit opportunities for cross contamination to occur.

Organics staff is authorized to retrieve samples from these storage areas for analysis. When analysis is complete, the samples are placed back into storage.

- 9.10 Sample Login.** Following sample custody transfer to the laboratory, the documentation that describes the clients analytical requirements are delivered to the sample login group for coding and entry to the Laboratory Information Management System (LIMS). This process translates all information related to collection time, turnaround time, sample analysis, and deliverables into a code which enables client requirements to be electronically distributed to the various departments within the laboratory for scheduling and execution. The technical staff is alerted to client or project specific requirements through the use of commenting in the LIMS.
- 9.11 Sample Retrieval for Analysis.** Retrieval priorities are established by the requesting department and submitted to the sample custodian when multiple requests are submitted. After sample analysis has been completed the analyst returns the sample to the storage area.
- 9.12 Sample Disposal.** Accutest retains all samples and sample extracts under proper storage for a maximum of 30 days following receipt. Longer storage periods are accommodated on a client specific basis if required. Samples may also be returned to the client for disposal.

Accutest disposes of all laboratory wastes following the requirements of the Resource Conservation and Recovery Act (RCRA). The Company has obtained and maintains an EPA waste generator identification number, CAL00218798

Sample management generates a sample disposal dump sheet from the LIMS tracking system as needed, which lists all samples whose holding period has expired.

Samples classified as PCB hazardous wastes are labeled and packaged according to the requirements in 40 CFR 761.

Laboratory wastes are collected by waste stream in designated areas throughout the laboratory. Waste streams are consolidated weekly and transferred to stream specific drums for disposal through a permitted waste management contractor.

All solvent extracts and digestates are collected for disposal following the thirty-day holding period and drummed according to their specific waste stream category. Chlorinated solvent extracts are drummed as chlorinated wastes (i.e., Methylene Chloride). Non-chlorinated solvent extracts are drummed as non-chlorinated wastes (i.e., acetone, hexane, methanol, and mixed solvents). Digestates are collected for disposal following the thirty-day holding period and drummed as corrosive liquid containing metals.

## 10.0 LABORATORY INSTRUMENTATION AND MEASUREMENT STANDARDS

**Requirement:** The laboratory has established procedures, which assure that instrumentation is performing to a pre-determined operational standard prior to the analysis of any samples. In general, these procedures follow the regulatory agency requirements established in promulgated methodology. The instrumentation selected to perform specified analysis are capable of providing the method specified uncertainty of measurement needed. These procedures are documented and incorporated into the standard operating procedures for the method being executed.

**10.1 Mass Tuning – Mass Spectrometers.** The mass spectrometer tune and sensitivity is monitored to assure that the instrument is assigning masses and mass abundances correctly and that the instrument has sufficient sensitivity to detect compounds at low concentrations. This is accomplished by analyzing a specific mass tuning compound at a fixed concentration. If the sensitivity is insufficient to detect the tuning compound, corrective action must be performed prior to the analysis of standards or samples. If the mass assignments or mass abundances do not meet criteria, corrective action must be performed prior to the analysis of standards or samples.

**10.2 Wavelength Verification – Spectrophotometers.** Spectrophotometer detectors are checked on a regular schedule to verify proper response to the wavelength of light needed for the test in use. If the detector response does not meet specifications, corrective action (detector adjustment or replacement) is performed prior to the analysis of standards or samples.

**10.3 Inter-element Interference Checks (Metals).** Inductively Coupled Plasma Emission Spectrophotometers (ICP) are subject to a variety of spectral interferences, which can be minimized or eliminated by applying interfering element correction factors and background correction points. Interfering element correction factors are checked on a specified frequency through the analysis of check samples containing high levels of interfering elements. Analysis of single element interferant solutions is also conducted at a specified frequency.

If the check indicates that the method criteria have not been achieved for any element in the check standard, the analysis is halted and data from the affected samples are not reported. Sample analysis is resumed after corrective action has been performed and the correction factors have been re-calculated.

New interfering element correction factors are calculated and applied whenever the checks indicate that the correction factors are no longer meeting criteria. At a minimum, correction factors are replaced once a year.

- 10.4 **Calibration and Calibration Verification.** Many tests require calibration using a series of reference standards to establish the concentration range for performing quantitative analysis. Instrument calibration is performed using standards that are traceable to national standards. Method specific procedures for calibration are followed prior to any sample analysis.

Calibration is performed using a linear regression calculation, calibration factors calculated from the curve, or other curve fitting equations. The calibration must meet method specific criteria for linearity or precision. If the criteria are not achieved, corrective action (re-calibration or instrument maintenance) is performed. The instrument must be successfully calibrated before analysis of samples can be conducted.

Initial calibration for metals analysis performed using inductively coupled plasma (ICP) employs the use of a single standard and a calibration blank to establish linearity.. The calibration blank contains all reagents that are placed into the calibration standard with the exception of the target elements. Valid calibration blanks must not contain any target elements.

Initial calibrations must be verified using a single concentration calibration standard from a second source. Second source standard is defined as a standard from a second or separate vendor. If no other vendor is available; then a second and separately prepared lot number from the primary vendor will suffice. The continuing validity of existing calibrations must be regularly verified using a single calibration standard. The response to the standard must meet pre-established criteria that indicate the initial calibration curve remains valid. If the criteria are not achieved corrective action (re-calibration) is performed before any additional samples may be analyzed.

Calibration verification is also performed whenever it appears that the analytical system is out of calibration or no longer meets the calibration requirements. It is also performed when the time period between calibration verifications has expired.

- 10.5 **Linear Range Verification and Calibration (ICP).** Linear range verification is performed for all ICP instrumentation. The regulatory program or analytical method specifies the verification frequency. A series of calibration standards are analyzed over a broad concentration range. The data from these analyses are used to determine the valid analytical range for the instrument. ICP instrument calibration is routinely performed using a single standard at a concentration within the linear range and a blank.

Some methods or analytical programs require a low concentration calibration check to verify that instrument sensitivity is sufficient to detect target elements at the reporting limit. The analytical method or regulatory program defines the criteria used to evaluate the low concentration calibration check. If the low calibration check fails criteria, corrective action is performed and verified through reanalysis of the low concentration calibration check before continuing with the field sample analysis. .

- 10.6 **Retention Time Development and Verification (GC)**. Chromatographic retention time windows are developed for all analysis performed using gas chromatographs with conventional detectors. An initial experimental study is performed, which establishes the width of the retention window for each compound. The retention time width of the window defines the time ranges for elution of specified target analytes on the primary and confirmation columns. Retention time windows are established upon initial calibration, applying the retention time range from the initial study to each target compound. Retention times are regularly confirmed through the analysis of an authentic standard during calibration verification. If the target analytes do not elute within the defined range during calibration verification, the instrument must be recalibrated and new windows defined. New studies are performed when major changes, such as column replacement are made to the chromatographic system.
- 10.7 **Equipment List**. See Appendix IV for a listing of all equipment used for measurement and/or calibration in laboratory processes.

## 11.0 INSTRUMENT MAINTENANCE

**Requirement.** Documented procedures have been established for conducting equipment maintenance. The procedure includes maintenance schedules if required or documentation of daily maintenance activities. All instrument maintenance activities are documented in instrument specific logbooks.

11.1 **Routine, Daily Maintenance.** Routine, daily maintenance is required on an instrument specific basis and is performed each time the instrument is used. Daily maintenance includes activities to insure a continuation of good analytical performance. This may include performance checks that indicate if non-routine maintenance is needed. If performance checks indicate the need for higher level maintenance, the equipment is taken out of service until maintenance is performed. Analysis cannot be continued until all performance checks meet established criteria and a return to operational control has been demonstrated and documented. The individual assigned to the instrument is responsible for daily maintenance.

11.2 **Non-routine Maintenance.** Non-routine maintenance is initiated for catastrophic occurrences such as instrument failure. The need for non-routine maintenance is indicated by failures in general operating systems that result in an inability to conduct required performance checks or calibration. Equipment in this category is taken out of service, tagged accordingly and repaired before attempting further analysis. Before initiating repairs, all safety procedures for safe handling of equipment during maintenance, such as lock-out/tag-out are followed. Analysis is not resumed until the instrument meets all operational performance checks criteria, is capable of being calibrated and a return to operational control has been demonstrated and documented. Section supervisors are responsible for identifying non-routine maintenance episodes and initiating repair activities to bring the equipment on-line. This may include initiating telephone calls to maintenance contractors if necessary. They are responsible for documenting all details related to the occurrence and repair.

11.3 **Scheduled Maintenance.** Modern laboratory instrumentation rarely requires regular preventative maintenance. If required, the equipment is placed on a schedule, which dictates when maintenance is needed. Examples include annual balance calibration by an independent provider or ICP preventative maintenance performed by the instrument manufacturer. Section supervisors are responsible for initiating scheduled maintenance on equipment in this category. Scheduled maintenance is documented using routine documentation practices.

11.4 **Maintenance Documentation.** Non-routine maintenance activities are documented in logbooks assigned to instruments and equipment used for analytical measurements. The logbooks contain preprinted forms, which specify the required maintenance activities. The analyst or supervisor performing or initiating the maintenance activity is required to check the activity upon its completion and initial the form. This includes documenting that the instrument has been returned to operational control following the completion of the activity.

## 12.0 QUALITY CONTROL PARAMETERS, PROCEDURES, AND CORRECTIVE ACTION

**Requirement.** All procedures used for test methods incorporate quality control parameters to monitor elements that are critical to method performance. Each quality parameter includes acceptance criteria that have been established by regulatory agencies for the methods in use. Criteria may also be established through the accumulation and statistical evaluation of internal performance data. Data obtained for these parameters during routine analysis must be evaluated by the analyst, and compared to the method criteria in use. If the criteria are not achieved, the procedures must specify corrective action and conformation of control before proceeding with sample analysis. QC parameters, procedures, and corrective action must be documented within the standard operating procedures for each method. In the absence of client specific objectives the laboratory must define qualitative objectives for completeness and representativeness of data.

**12.1 Procedure.** Bench analysts are responsible for methodological quality control and sample specific quality control. Each method specifies the control parameters to be employed for the method in use and the specific procedures for incorporating them into the analysis. These control parameters are analyzed and evaluated with every designated sample group (batch).

The data from each parameter provides the analyst with critical decision making information on method performance. The information is used to determine if corrective action is needed to bring the method or the analysis of a specific sample into compliance. These evaluations are conducted throughout the course of the analysis. Each control parameter is indicative of a critical control feature. Failure of a methodological control parameter is indicative of either instrument or batch failure. Failure of a sample control parameter is indicative of control difficulties with a specific sample or samples.

**Sample Batch.** All samples analyzed in the laboratory are assigned to a designated sample batch, which contains all required quality control samples and a defined maximum number of field samples that are prepared and/or analyzed over a defined time period. The maximum number of field samples in the batch is 20. ANC has incorporated the NELAP batching policy as the sample-batching standard. This policy incorporates the requirement for blanks and spiked blanks as a time based function as defined by NELAP. Accordingly, the specified time period for a sample batch is 24 hours. Matrix spike/matrix spike duplicate, matrix spikes and duplicates are defined as sample frequency based functions and may be applied to several batches until the frequency requirement has been reached. A matrix spike/matrix spike duplicate, matrix spikes and/or duplicate is required every 20 samples.

Client criteria that defines a batch as a time based function which includes a matrix spike/matrix spike duplicates as a contractual specification will be honored. The typical batch contains a blank and a laboratory control sample (LCS or spiked blank). Batch documentation includes lot specifications for all reagents and standards used during preparation of the batch.

**12.2 Methodological Control Parameters and Corrective Action.** Prior to the analysis of field samples the analyst must determine that the method is functioning properly. Specific control parameters indicate whether critical processes meet specified requirements before continuing with the analysis. Method specific control parameters must meet criteria before sample analysis can be conducted. Each of these parameters is related to processes that are under the control of the laboratory and can be adjusted if out of control.

**Method Blank.** A method blank is analyzed during the analysis of any field sample. The method blank is defined as a sample. It contains the same standards (internal standards, surrogates, matrix modifiers, etc.) and reagents that are added to the field sample during analysis, with the exception of the sample itself. If the method blank contains target analyte(s) at concentrations that exceed reporting limit concentrations and is greater than 1/10 of the amount measured in any sample, the source of contamination is investigated and eliminated before proceeding with sample analysis. Systematic contamination is documented for corrective action and resolved following the established corrective action procedures.

**Blank Spikes (LCS).** A laboratory control sample (spiked blank or commercially prepared performance evaluation sample) is analyzed along with field samples to demonstrate that method accuracy is within acceptable limits. These spike solutions may be from different sources than the sources of the solutions used for method calibration depending upon the method requirements. The performance limits are derived from published method specifications or from statistical data generated from the analysis of laboratory method performance samples. Spiked blanks are blank matrices (reagent water or clean sand) spiked with target parameters and analyzed using the same methods used for samples. Accuracy data is compared to laboratory derived limits to determine if the method is in control. Laboratory control samples (LCS) are commercially prepared spiked samples in an inert matrix. Performance criteria for recovery of spiked analytes are pre-established by the commercial entity preparing the sample. The sample is analyzed in the laboratory as an external reference.

Accuracy data is compared to the applicable performance limits. If the spike accuracy exceeds the performance limits, corrective action, as specified in the SOP for the method is performed and verified before continuing with a field sample analysis.

Blanks and spikes are routinely evaluated before samples are analyzed. However, in situations where sample analysis is performed using an autosampler, they may be evaluated after sample analysis has occurred. If the blanks and spikes do not meet criteria, sample analysis is repeated.

**Proficiency Testing.** Proficiency test samples (PTs) are single or double blind spikes, introduced to the laboratory to assess method performance. PTs may be introduced as double blinds submitted by commercial clients, single or double blinds from regulatory agencies, or internal blinds submitted by the QA group.

A minimum of two single blind studies must be performed each year for every parameter in aqueous and solid matrices for each field of testing for which the laboratory maintains

accreditation. Proficiency samples must be purchased as blinds from an A2LA accredited vendor. Data from these studies are provided to the laboratory by the vendor and reported to accrediting agencies. If unsatisfactory performance is noted, corrective action is performed to identify and eliminate any sources of error. A new single blind must be analyzed if required to demonstrate continuing proficiency.

PT samples performed for accrediting agencies or clients, which do not meet performance specifications, require a written summary that documents the corrective action investigation, findings, and corrective action implementation.

Single or double blind proficiency test samples may be employed for self-evaluation purposes. Data from these analyses are compared to established performance limits. If the data does not meet performance specifications, the system is evaluated for sources of acute or systematic error. If required, corrective action is performed and verified before initiating or continuing sample analysis.

**Trend Analysis for Control Parameters.** The quality assurance staff is responsible for continuous analytical improvement through quality control data trend analysis. Accuracy data for spiked parameters in the spiked blank are statistically evaluated daily for trends indicative of systematic problems. Data from LCS parameters and surrogates are pooled on a method, matrix, and instrument basis. This data is evaluated by comparison to existing control and warning limits. Trend analysis is performed automatically as follows:

- Any point outside the control limit
- Any three consecutive points between the warning and control limits
- Any eight consecutive points on the same side of the mean.
- Any six consecutive points increasing or decreasing

**12.3 *Sample Control Parameters and Corrective Action.*** The analysis of samples can be initiated following a successful demonstration that the method is operating within established controls. Additional controls are incorporated into the analysis of each sample to determine if the method is functioning within established specifications for each individual sample. Sample QC data is evaluated and compared to established performance criteria. If the criteria are not achieved the method or the SOP specifies the corrective action required to continue sample analysis. In many cases, failure to meet QC criteria is a function of sample matrix and cannot be remedied. Each parameter is designed to provide quality feedback on a defined aspect of the sampling and analysis episode.

**Duplicates.** Duplicate sample analysis is used to measure analytical precision. This can also be equated to the samples homogeneity. Precision criteria are method dependent. If precision criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

**Laboratory Spikes & Spiked Duplicates.** Spikes and spiked duplicates are used to measure analytical precision and accuracy for the sample matrix selected. Precision and accuracy criteria are method dependent. If precision and accuracy criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before reporting sample data.

**Serial Dilution (Metals).** Serial dilutions of metals samples are analyzed to determine if analytical matrix effects may have impacted the reported data. If the value of the serially diluted samples does not agree with the undiluted value within a method-specified range, the sample matrix may be causing interferences, which may lead to either a high or low bias. If the serial dilution criterion is not achieved, it must be flagged to indicate possible bias from matrix effects.

**Post Digestion Spikes.** Digested samples are spiked and analyzed to determine if matrix interferences are biasing the results when the pre-digestion spike (matrix spike) recovery falls outside the control limits. It may also be used to determine potential interferences per client's specification. The sample is spiked at the concentration specified in the method SOP. No action is necessary if the post digestion spike is outside of the method criteria, unless a preparation problem is suspected with the spike, in which case the post digestion spike should be re-prepared and reanalyzed.

**Surrogate Spikes (Organics).** Surrogate spikes are organic compounds that are similar in behavior to the target analytes but unlikely to be found in nature. They are added to all quality control and field samples to measure method performance for each individual sample. Surrogate accuracy limits are derived from published method specifications or from the statistical evaluation of laboratory generated surrogate accuracy data. Accuracy data is compared to the applicable performance limits. If the surrogate accuracy exceeds performance limits, corrective action, as specified in the method or SOP is performed before sample data can be reported.

**Internal Standards (Organic Methods).** Internal standards are retention time and instrument response markers added to every sample to be used as references for quantitation. Their response is compared to reference standards and used to evaluate instrument sensitivity on a sample specific basis. Internal standard retention time is also compared to reference standards to assure that target analytes are capable of being located by their individual relative retention time.

If internal standard response criteria are not achieved, corrective action or additional action may be required. The recommended action must be completed before sample data can be reported.

If the internal standard retention time criteria are not achieved corrective action or additional action may be required. This may include re-calibration and re-analysis. Additional action must be completed before sample data is reported.

- 12.4 **Laboratory Derived Quality Control Criteria.** Control criteria for in-house methods and client specific modifications that exceed the scope of published methodology are defined and documented prior to the use of the method. The Quality Assurance Director is responsible for identifying additional control criteria needs. Control parameters and criteria, based on best technical judgment are established using input provided by the operations staff. These control parameters and criteria are documented and incorporated into the method.

The laboratory-derived criteria are evaluated for technical soundness on spiked samples prior to the use of the method on field samples. The technical evaluation is documented and archived by the Quality Assurance Staff.

When sufficient data from the laboratory developed control parameter is accumulated, the data is statistically processed and the experimentally derived control limits are incorporated into the method.

- 12.5 **Bench Review & Corrective Action.** The bench chemists are responsible for all QC parameters. Before proceeding with sample analysis, they are required to successfully meet all instrumental QC criteria. They have the authority to perform any necessary corrective action before proceeding with sample analysis. Their authority includes the responsibility for assuring that departures from documented policies and procedures do not occur.

The bench chemists are also responsible for all sample QC parameters. If the sample QC criteria are not achieved, they are authorized and required to perform the method specified corrective action before reporting sample data.

- 12.6 **Data Qualifiers.** An alpha character coding system is employed for defining use limitations for reported data. These limitations are applied to analytical data by the analyst to clarify the usefulness of the reported data for data user. Common data qualifiers and their definitions are as follows:

**Organics.**

- J: Indicates an estimated value. Applied to calculated concentrations for tentatively identified compounds and qualitatively identified compounds whose concentration is below the reporting limit, but above the MDL.
- N: Indicates qualitative evidence of a tentatively identified compound whose identification is based on a mass spectral library search and is applied to all TIC results.
- C: Applied to pesticide data that has been qualitatively confirmed by GC/MS.
- B: Used for analytes detected in the sample and its associated method blank.
- E: Applied to compounds whose concentration exceeds the upper limit of the calibration range.

**Metals and Inorganics.**

- J: Applied if the reported concentration value was less than the reporting limit but greater than the MDL.
- E: Estimated concentration caused by the presence of interferences, normally applied when the serial dilution is out.
- \*\*\*: Spike sample recovery not within control limits, or Duplicate or matrix spike duplicate analysis not within control limits.

**12.7 QA Monitoring.** The Reporting staff conducts a spot review of completed data packages prior to client release for specified projects. This review includes an examination of QC data for compliance. If non-conformances are detected, the reporting staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation. The data package is released when the package becomes compliant with all quality requirements. If compliance is not possible, the data is qualified and an appropriate case narrative is generated for inclusion in the data package.

If the review reveals trends indicative of systematic problems, QA initiates an investigation to determine the cause. If process defects are detected, a corrective action is implemented and monitored for effectiveness.

**Performance Limits.** The Quality Assurance Director is responsible for compilation and maintenance of all precision and accuracy data used for performance limits. Quality control data for all test methods are accumulated and stored in the laboratory information management system (LIMS). Parameter specific QC data is extracted annually and statically processed to develop laboratory specific warning limits and control limits. The new limits are reviewed and approved by the supervisory staff prior to their use for data assessment. The new limits are used to evaluate QC data for compliance with method requirements for a period of one year. Laboratory generated limits appear on all data reports.

**12.8 Data Package Review.** Accutest employs multiple levels of data review to assure that reported data has satisfied all quality control criteria and that client specifications and requirements have been met. Each production department has developed specific data review procedures, which must be completed before data is released to the client.

**Analytical Review.** The analyst conducts the primary review of all data. This review begins with a check of all instrument and method quality control and progresses through sample quality control, concluding with a check to assure that the client's requirements have been executed. Analyst checks focus on a review of qualitative determinations and checks of precision and accuracy data to verify that existing laboratory criteria have been achieved. Checks at this level may include comparisons with project specific criteria if applicable. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.

Analysts who have met the qualification criteria for the method in use perform secondary, peer level data reviews. Analyst qualification requirements include a valid demonstration of capability and demonstrated understanding of the method SOP. Section supervisors may perform secondary review in-lieu of a peer review. Supervisors or qualified designees review 100% of the data produced by their department. It includes a check of all manual calculations; an accuracy check of manually transcribed data from bench sheets to the LIMS, a check of calibration and continuing calibration, all QC criteria and a comparison of the data package to client specified requirements. Also included are checks to assure the appropriate methodology was applied and that all anomalous information was properly flagged for communication in the case narrative. Supervisors have the authority to reject data and initiate re-analysis, corrective action, or reprocessing.

All laboratory data requiring manual entry into LIMS system is double-checked by the analysts performing initial data entry and the section reviewer. Verification of supervisory review is indicated on the raw data summary by the reviewer's initials and date.

Hard copies of manually integrated chromatographic peaks are printed that clearly depict the manually drawn baseline. The hard copy is reviewed and approved by the section supervisor (initialed and dated) and included in the data package of all full tier reports or the archived batch records of commercial report packages.

A manager or supervisor only has permission to edit electronic data that has been committed to the LIMS. These edits may be required if needs for corrections are indicated during the final review. A GALP audit record for all electronic changes in the LIMS is automatically appended to the record.

The group manager performs a tertiary review on a spot check basis. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

**Report Generation Review.** The report generation group reviews all data and supporting information delivered by the laboratory for completeness and compliance with client specifications. Missing deliverables are identified and obtained from the laboratory. The group also reviews the completed package to verify that the delivered product complies with all client specifications. Non-analytical defects are corrected before the package is sent to the client.

**Project Management/Quality Control Review.** Spot-check data package reviews are performed by the project management staff. Project management reviews focus on project specifications. If the project manager identifies defects in the product prior to release, he initiates immediate corrective action to rectify the situation.

**Data Reporting.** Analytical data is released to clients following a secondary review by the group supervisor. Hard copy support data is compiled by the report generation group and

assembled into the final report. The report is sent to the client following reviews by the report generation staff.

All data reports include specified information, which is required to identify the report and its contents. This information includes a title, name and address of the laboratory, a unique report number, total number of pages, clients name and address, analytical method identification, arriving sample condition, sample and analysis dates, test results with units of measurement, authorized signature of data release, and NELAC requirements certification.

- 12.9** **Electronic Data Reduction.** Raw data from sample analysis is entered into the laboratory information management system (LIMS) using automated processes or manual entry. Final data processing is performed by the LIMS using procedures developed by the Company.

All LIMS programs are tested and validated prior to use to assure that they consistently produce correct results. The Information Technology Staff performs software validation testing. The testing procedures are documented in an SOP. Software programs are not approved for use until they have demonstrated that they are capable of performing the required calculations.

- 12.10** **Representativeness.** Data representativeness is based on the premise that qualitative and quantitative information developed for field samples is characteristic of the sample that was collected by the client and analyzed in the laboratory. The laboratory objective for representativeness defines data as representative if the criteria for all quality parameters associated with the analysis of the sample are achieved.

- 12.11** **Comparability.** Analytical data is defined as comparable when data from a sample set analyzed by the laboratory is representatively equivalent to other sample sets analyzed separately regardless of the analytical logistics. The laboratory will achieve 100% comparability for all sample data which meets the criteria for the quality parameters associated with its analysis using the method requested by the client.

### 13.0 CORRECTIVE ACTION SYSTEM (Incident Reporting)

**Requirement.** The laboratory employs a procedure for correcting defective processes, safety issues, efficiency issues, systematic errors, and quality defects enabling the staff to systematically improve product quality. The system includes procedures for communicating items requiring corrective action to responsible individuals, corrective action tracking procedures, corrective action documentation, monitoring of effectiveness, and reports to management. The system is fully documented in a standard operating procedure. Individual corrective actions and responses are documented in a dedicated database.

- 13.1 **Procedure.** Corrective action is the step that follows the identification of a process defect. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance. The formal system is maintained by the quality assurance department. Operations management is responsible for working within the system to resolve identified deficiencies.

**Routine Corrective Action.** Routine corrective action is defined as the procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters or analytical system specifications. Bench analysts have full responsibility and authority for performing routine corrective action. The resolution of defects at this level does not require a procedural change or staff re-training. The analyst is free to continue work once corrective action is complete and the analytical system has been returned to control. Documentation of routine corrective actions is limited to logbook comments for the analysis being performed.

**Non-routine corrective action.** Non-routine corrective action applies to situations where the bench analysts failed to perform routine corrective action before continuing analysis. Supervisors and Department Managers perform corrective action in these situations. Documentation of all non-routine corrective actions is performed using the corrective action system.

**Turnbacks.** Turnbacks are defined as anything that stops your work, but does not affect quality. These efficiency issues are tracked in the incident reporting system, but do not need immediate corrective action. Repeat turnbacks may result in their status changing to a corrective action worthy of Supervisor/Manager attention and a process change. Process change requires development, documentation, planning, implementation and training.

- 13.2 **Documentation & Communication.** Routine corrective actions are documented as part of the analytical record. Notations are made in the comments section of the analytical chronicle or data sheet detailing the nonconformance and corrective action. Continuation of the analysis indicates that return to control was successful.

Corrective actions for process changes are documented, tracked and monitored for effectiveness. Supervisors or senior staff members may initiate corrective actions by generating a Corrective Action Report.

The Corrective Action System is an MSAccess application designed by our IT Dept. Authorized staff have a short-cut on their computer desk-top to the Corrective Action Database. The initiator fills out the information and a Corrective Action Report is sent to the responsible party and others as necessary via E-mail. The CAR is reviewed responded to via the database. Corrective Actions are numbered and tracked by the QA Manager.

The responsible party identifies the root cause of the defect, initiates the immediate fix and develops and implements the procedural change. Existing documentation such as SOPs are edited to reflect the change. The affected staff is informed of the procedural change through a formal training session. The training is documented and copies are placed into individual training files. The corrective action form is completed by the responsible party and returned to the QA Manager. All relevant information for the CAR is stored in the appropriate directory by the assign number.

**Monitoring.** The QA Staff monitors the implemented corrective action until it is evident that the action has been effective and the defect has been eliminated. The corrective actions are updated by QA to reflect closure of the corrective action. The QA staff assigns a number to the corrective action tracking. Additional monitoring of the corrective action may be conducted during routine laboratory audits.

If QA determines that the corrective action response has not effectively remedied the deficiency, the process continues with a re-initiation of the corrective action. Corrective action continues until the defect is eliminated. If another procedural change is required, it is treated as a new corrective action, which is documented and monitored using established procedures.

**Client Notification.** Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, ANC will notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

## 14.0 PROCEDURES FOR EXECUTING CLIENT SPECIFICATIONS

**Requirement.** Systems have been established for evaluating and processing client specifications for routine and non-routine analytical services. The systems enable the client services staff to identify, evaluate, and document the requested specifications to determine if adequate resources are available to perform the analysis. The system includes procedures for communicating the specifications to the laboratory staff for execution and procedures for verifying the specifications have been executed.

- 14.1 **Client Specific Requirements.** The project manager is the primary contact for clients requesting laboratory services. Client specifications are communicated using several mechanisms. The primary sources of information are the client's quality assurance project plan (QAPP) and the analytical services contract both of which detail the analytical, quality control and data reporting specifications for the project. In the absence of a QAPP, projects specifications can also be communicated using contracts, letters of authorization, or letters of agreement, which may be limited to a brief discussion of the analytical requirements and the terms and conditions for the work. These documents may also include pricing information, liabilities and scope of work, in addition to the analytical requirements. QAPPs include detailed analytical requirements and data quality objectives, which supersede those found in the referenced methods. This information is essential to successful project completion.

The client services staff provides additional assistance to clients who are unsure of the specifications they need to execute the sampling and analysis requirements of their project. They provide additional support to clients who require assistance in results interpretation as needed, provided they possess the expertise required to render an opinion.

The project manager is responsible for obtaining project documents, which specify the analytical requirements. Following project management review, copies are distributed to the QA Director and the appropriate departmental managers for review and comment. The original QAPP is filed in a secure location.

- 14.2 **Requirements for Non-Standard Analytical Specifications.** Client requirements that specify departures from documented policies, procedures, or standard specifications must be submitted to Accutest in writing. These requirements are reviewed and approved by the technical staff before the project is accepted. Once accepted, the non-standard requirements become analytical specifications, which follow the routine procedure for communicating client specifications. Specific Client Specifications are filed in the Project Management Directory. Departures from documented policies, procedures, or standard specifications that do not follow this procedure are not permitted.

- 14.3 Evaluation of Resources.** A resource evaluation is completed prior to accepting projects submitted by clients. The evaluation is initiated by the client services staff who prepares a brief synopsis that includes the logistical requirements of the project. Logistical specifications for new projects are summarized in writing for evaluation by the affected departments. The specifications are evaluated by the department manager from a scheduling and hardware resources perspective. The project is not accepted unless the department managers have the necessary resources to execute the project according to client specifications.
- 14.4 Documentation.** New projects are initiated using a project set up form, which is completed prior to the start of the project. This form details all of the information needed to correctly enter the specifications for each client sample into the laboratory information management system (LIMS). The form includes data reporting requirements, billing information; data turnaround times, QA level, state of origin, and comments for detailing project specific requirements. The project manager is responsible for obtaining this information from the client and completing the form prior to sample arrival and login. For less complicated requirements or unscheduled work; the chain of custody will contain all the necessary information.

Sample receipt triggers project creation and the login process. The information on the set-up form is entered into the LIMS immediately prior to logging in the first sample. The set up form may be accompanied by a quotation, which details the analytical product codes and sample matrices. These details are also entered into the LIMS during login.

Special information is distributed to the laboratory supervisors and login department in electronic or hardcopy format upon project setup. All, project specific information is retained on the file server.

Job Level Documentation:

\\ancdoc\Documents\Disk1\coc

Account and Project Level Documentation

\\Accunca.accutest.com\depts\project\_mgt

All client communications; regarding a job, project or account is documented either via email and notes stored as .pdfs in the directories above.

Department managers prepare summary sheets that detail client specific analytical requirements for each test. Bench analysts use these sheets to obtain information regarding client specific analytical requirements before analyzing samples. A program code is established for each client that links the client specifications to a client project. This code is attached to a project by the project manager at login and listed on the work list for each work group conducting analysis for clients with standing requirements.

- 14.5 Communication.** A pre-project meeting is held between client services and the operations managers to discuss the specifications described in the QAPP, contract and/or related

documents. Project logistics are discussed and finalized and procedures are developed to assure proper execution of the client's analytical specifications and requirements. Questions, raised in the review meeting, are discussed with the client for resolution. Exceptions to any requirements, if accepted by the client, are documented and incorporated into the QAPP or project documentation records.

Non-standard specifications for individual clients are documented in the LIMS at the client account level or program level. Simple specifications are documented as comments for each project. Once entered into the LIMS, these specifications become memorialized for all projects related to the client account. Complex specifications are assigned program codes that link the specification to detailed analytical specifications.

Specifications that are not entered into the LIMS are prohibited unless documented in an interdepartmental memo, which clearly identifies the project, client and effective duration of the specification.

- 14.6** **Operational Execution.** A work schedule (WIP) is prepared for each analytical department on a daily basis. Analytical specifications or program codes from recently arrived samples have now been entered into the LIMS database. The database is sorted by analytical due date and holding time, into product specific groups. Samples are scheduled for analysis by due date and holding time. The completed schedule, which is now defined as a work list, is printed. The list contains the client requested product codes, program codes and specifications required for the selected sample(s). Special requirements are communicated to the analyst using the comments section or relayed through verbal instructions provided by the supervisor. The bench analyst assumes full responsibility for performing the analysis according to the specifications printed on the work sheet.
- 14.7** **Verification.** Prior to the release of data to the client, laboratory section managers and the report generation staff review the report and compare the completed product to the client specifications documentation to assure that all requirements have been met. Project managers perform a spot check of projects with unique requirements to assure that the work was executed according to specifications.

## 15. CLIENT COMPLAINT RESOLUTION PROCEDURE

**Requirement.** The laboratory uses the Incident Reporting System to investigate and manage client complaints. The system includes procedures for documenting the complaint and communicating it to the appropriate department for resolution. The system also includes a quality assurance evaluation to determine if the complaint is related to systematic defects requiring corrective action and process changes.

- 15.1 Procedure.** Client complaints are entered into the Incident Report System by any member of the staff. The report is sent directly to the Laboratory Director and the QA Officer. Reports are assigned to the responsible departments for resolution. The resolution is reviewed by quality assurance (QA) and the originator, then communicated to the client. QA reviews the complaint and resolution to determine if systematic defects exist. If no systemic defects are present and the proposed resolution is sufficient, QA will close the complaint/inquiry with a No Further Action necessary. If systematic defects are present, QA initiates an additional incident report for the responsible party who develops and implements a response that eliminates the defect.
- 15.2 Documentation.** A record of conversation is maintained within the PDF Incident Report. The message is distributed to the QA staff and the party bearing responsibility for resolution by E-Mail. The complaint resolution is documented on the message by the responsible party and returned to the originator. A copy is sent to QA for review.
- 15.3 Corrective Action.** Responses to data queries are required from the responsible party. At a minimum, the response addresses the query and provides an explanation to the complaint. Formal corrective action may focus on the single issue expressed in the complaint. Corrective action may include reprocessing of data, editing of the initial report, and re-issue to the client. If the QA review indicates a systematic error, process modification is required. The defective process at the root of the complaint is changed. SOPs are either created or modified to reflect the change. The party responsible for the process implements process changes.
- 15.4 QA Monitoring.** Process changes, implemented to resolve systematic defects, are monitored for effectiveness by QA. If monitoring indicates that the process change has not resolved the defect, QA works with the department management to develop and implement an effective process. If monitoring indicates that the defect has been resolved, monitoring is slowly discontinued and the corrective action is closed. Continued monitoring is incorporated as an element of the annual system audit.

## 16.0 CONTROL OF NONCONFORMING PRODUCT

**Requirement:** Policies and procedures have been developed and implemented that describe the procedures employed by the laboratory when any aspect of sample analysis or data reporting do not conform to established procedures or client specifications. These procedures include steps to ensure that process defects are corrected and affected work is evaluated to assess its impact to the client. Non-conforming issues are also handled in the Incident Reporting system.

**16.1 Procedure:** Nonconforming product is identified through routine internal review and audit practices or through client inquiry. The individuals who identify the nonconformance or receiving a nonconformance inquiry immediately initiate an Incident Report informing the Laboratory Director and the Quality Assurance Director. The Laboratory Director initiates an evaluation of the nonconformance through the Quality Assurance Department and takes full responsibility for managing the process and identifying the course of action to take, initiating corrective action and mitigating the impact of the nonconformance to the client.

**16.2 Corrective Action:** The outcome of the evaluation dictates the course of action. This includes client notification when the quality of data reported has been impacted and may also include corrective action if applicable. However, additional action may be required including cessation of analysis and withholding and or recalling data reports. If the evaluation indicates that nonconforming data may have been issued to clients, the client is immediately notified and data may be recalled. If work has been stopped because of a nonconformance, the Laboratory Director is the only individual authorized to direct a resumption of analysis.

Nonconformance caused by systematic process defects require retraining of the personnel involved as an element of the corrective action solution.

**16.3 Date Inquiry Program:** A program contained within the Corrective action program used to capture, address, and respond to client challenges to our sample results or data package components. A Complaint or Data Inquiry is begun by using the Initiate icon. This leads to an interactive template which allows the user to enter an Accutest Job number if it is a data inquiry. If it is a complaint unrelated to an Accutest job, the program will open directly to the template requiring the user to populate any number of the program fields. If using the Data Inquiry entry point along with an Accutest job number, the program will automatically populate some of the fields by extracting the information from LIMS.

## 17.0 CONFIDENTIALITY PROTECTION PROCEDURES

**Requirement:** Policies and procedures have been developed to protect client data from release to unauthorized parties or accidental release of database information through accidental electronic transmission or illegal intrusion. These policies have been communicated to clients and staff. Electronic systems are regularly evaluated for effectiveness.  
(SOP PM004) -Confidentiality Protection Procedures

- 17.1 **Client Anonymity.** Information related to the Company's clients is granted to employees on a "need to know" basis. An individual's position within the organization defines his "need to know". Individuals with "need to know" status are given password access to systems that contain client identity information and access to documents and document storage areas containing client reports and information. Access to client information by individuals outside of the Company is limited to the client and individuals authorized by the client.

Individuals outside of the Company may obtain client information through subpoena issued by a court of valid jurisdiction. Clients are informed when subpoenas are received ordering the release of their information.

Client information may be released directly to regulatory agencies without receiving client authorization under specified circumstances. These circumstances require that the regulatory agency have statutory authority under the regulations for laboratory certification and that ANC's operations fall under the purview of the regulation. In these situations, Accutest will inform the client of the regulatory agencies request for information pertaining to his data and proceed with the delivery of the information to the regulatory agency.

- 17.2 **Documents.** Access to client documents is restricted to employees in need to know positions. Copies of all client reports are stored in secure electronic archives with restricted access. Reports and report copies are distributed to individuals who have been authorized by the client to receive them. Data reports or data are not released to third parties without verbally expressed or written permission from the client.
- 17.3 **Electronic Data.**

**Database Intrusion.** Direct database entry is authorized for employees of Accutest only on a need to know basis. Entry to the database is restricted through a user specific multiple password entry system. Direct access to the database outside of the facility is possible through a dial-up connection. A unique password is required for access to the local area network. A second unique password is required to gain access to the database. The staff receives read or write level authorization on a hierarchical privilege basis.

**17.4 Information Requests.** Client specific data or information is not released to third parties without verbally expressed or written permission from the client. Written permission is required from third parties, who contact the Company directly for the release of information. Verbal requests will be honored only if they are received directly from the client. These requests must be documented in a record of communication maintained by the authorized recipient.

**17.5 Transfer of Records.** Archived data, which has previously been reported and transmitted to clients, is the exclusive property of Accutest Laboratories. In the event of a cessation of business activities due to business failure or sale, The Company's legal staff will be directed to arrange for the final disposition of archived data.

The final disposition of archived data will be accomplished using the approach detailed in the following sequence:

1. All data will be transferred to the new owners for the duration of the required archive period as a condition of sale.
2. If the new owners will not accept the data or the business has failed, letters will be sent to clients listed on the most recent active account roster offering them the option to obtain specific reports (identified by Accutest Job Number) at their own expense.
3. A letter will be sent to the NELAC accrediting authority with organizational jurisdiction over the company offering them the option to obtain all unclaimed reports at their own expense.
4. All remaining archived data will be recycled using the most expedient means possible.

## 18.0 QUALITY AUDITS AND SYSTEM REVIEWS

**Requirement:** The quality assurance group conducts regularly scheduled audits of the laboratory to assess compliance with quality system requirements, technical requirements of applied methodology, and adherence to documentation procedures. The information gathered during these audits is used to provide feedback to senior management and perform corrective action where needed for quality improvement purposes.

- 18.1 **Quality System Reviews.** Quality system reviews are performed annually by the Quality Assurance Director for the Company President. In this review, the laboratory is evaluated for compliance with the laboratory Quality Systems Manual (QSM) and the quality system standards of the National Environmental Laboratory Accreditation Conference. Findings, which indicate non-compliance or deviation from the QSM, are flagged for corrective action. Corrective actions require either a return to compliance or a plan change to reflect an improved quality process. The Quality Assurance Director is responsible for making and documenting changes to the QSM. These changes are reviewed by the Company President and The Laboratory Director prior to the approval of the revised system.
- 18.2 **Quality System Audits.** Quality system audits are conducted to evaluate the effectiveness and laboratory compliance with individual quality system elements. These audits are conducted on an established schedule. Audit findings are documented and communicated to the management staff and entered into the corrective action system for resolution. If necessary, retraining is conducted to assure complete understanding of the system requirements.
- 18.3 **Test Method Assessments.** Test Method Assessments are performed throughout the year following an established schedule. Selected analytical procedures are evaluated for compliance with standard operating procedures (SOPs) and method requirements. If non-conformances exist, the published method serves as the standard for compliance. SOPs are edited for compliance if the document does not reflect method requirements. Analysts are trained to the new requirements and the process is monitored by quality assurance. Analysts are retrained in method procedures if an evaluation of bench practices indicates non-compliance with SOP requirements.
- 18.4 **Documentation Audits.** Documentation audits are conducted annually. This audit includes a check of measurement processes that require manual documentation. It also includes checks of data archiving systems and a search to find and remove any inactive versions of SOPs that may still be present in the laboratory and being accessed by the analysts. Non-conformances are corrected on the spot. Procedural modifications are implemented if the evaluation indicates a systematic defect.
- 18.5 **Corrective Action Monitoring.** Defects or non-conformances that are identified during client or internal audits are documented in the corrective action systems and corrected through process modifications and/or retraining. Once a corrective action has been designed and

implemented, it is monitored for compliance on a regular basis by the QA staff. Spot corrections are performed if the staff is not following the new procedure. Monitoring of the corrective action continues until satisfactory implementation has been verified.

- 18.6 **Preventive Action.** Laboratory systems or processes, which may be faulty and pose the potential for nonconformances, errors, confusing reports or difficulties establishing traceability may be identified during internal audits. These items are highlighted for systematic change using the corrective action/incident reporting system and managed to .
- 18.7 **Client Notification.** Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, Accutest will immediately notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.
- 18.8 **Management Reports.** Formal reports of all audit and proficiency testing activity are prepared for the management staff and presented as they occur. Additional reports may be presented orally at regularly scheduled staff meetings

Management reports may also address the following topics:

- Status and results of internal and external audits,
- Status and results of internal and external proficiency testing,
- Identification of quality control problems in the laboratory,
- Discussion of corrective action/incident reporting program issues,
- Status of external certifications and approvals,
- Status of staff training and qualifications,
- Discussion of new quality system initiatives.
- Recommendations for further action on listed items are included in the report.

## 19.0 HEALTH AND SAFETY

**Requirement.** The company operates a formal health and safety program that complies with the requirements of the Occupational Health and Safety Administration. The program consists of key policies and practices that are essential to safe laboratory operation. All employees are required to receive training on the program elements. Job specific training is conducted to assure safe practices for specific tasks. All employees are required to participate in the program, receive initial and annual training, and comply with the program requirements. All plan and program requirements are detailed in the Health and Safety Program Manual.

- 19.1 Policy.** Accutest Laboratories – Northern California will provide a safe and healthy working environment for its employees and clients while protecting the public and preserving the Company's assets and property. The company will comply with all applicable government regulations pertaining to safety and health in the laboratory and the workplace.

The objective of the Accutest-Northern California Health and Safety Program is to promote safe work practices that minimize the occurrence of injuries and illness to the staff through proper health and safety training, correct laboratory technique application and the use of engineering controls.

- 19.2 Responsibilities.** The Health and Safety Program assists managers, supervisors and non-supervisory employees in control of hazards and risks to minimize the potential for employee and client injuries, damage to client's property and damage or destruction to ANC's facility.

The Health, Safety and Facilities Manager is responsible for implementing the Program's elements and updating its contents as necessary. He also conducts periodic audits to monitor compliance and assess the program's effectiveness. The Health, Safety and Facilities Manager is also responsible for creating and administering safety training for all new and existing employees.

The employee is responsible for following all safety rules established for their protection, the protection of others and the proper use of protective devices provided by the Company. The employee is also expected to comply with the requirements of the program at all times. Department Managers and Supervisors are responsible for ensuring the requirements of the Safety Program are practiced daily. The Company President retains the ultimate responsibility for the program design and implementation.

- 19.3 Program Elements.** The Accutest Health and Safety Program consist of key program elements that compliment the company's health and safety objective. These elements form the essence of the health and safety policy and assure that the objectives of the program are achieved.

***Safety Education and Training and Communication.*** Training is conducted to increase the staff's awareness of laboratory hazards and their knowledge of the safety practices and

procedures required to protect them from those hazards. It is also used to communicate general safety procedures required for safe operation in a chemical laboratory.

Initial health and safety training for new employees is conducted during orientation. The training focuses on the Accutest Safety and Health Program and includes specific training for the hazards that may be associated with the employees duties. Training is also conducted for all program elements focusing on general, acceptable, laboratory safety procedures. Targeted training is conducted to address hazards or safety procedures that are specific to individual employee's work assignments. All training activities are documented and archived in individual training folders, A health and safety training inventory is maintained in the training database.

***Safety Committee.*** The safety committee provides the employee with an opportunity to express their views and concerns on safety issues in a forum where those concerns will be addressed. This committee meets monthly to assure that the interests of the company and the well being of the employee are protected. They also serve as a catalyst for elevating the level of safety awareness among their peers.

***Hazard Identification and Communication.*** The hazard communication program enables employees to readily identify laboratory hazards and the procedures to protect themselves from those hazards. This program complies with OSHA's Hazard Communication Standard, Title 29 Code of Federal Regulations 1910.1200 that requires the company to adopt and adhere to the following key elements:

- ◆ Material Safety Data Sheets (MSDS) must be available to any employee wishing to view them,
- ◆ The Company must maintain a Hazardous Chemicals Inventory (by location), which is updated on an annual basis,
- ◆ Containers are properly labeled,
- ◆ All employees must be provided with annual Hazard Communication and Right to Know training,

The hazard communication program also complies with the requirements of the New Jersey Worker and Community Right to Know Law, NJAC 8:95.

***Identification of Workplace Hazards.*** The workplace hazard identification procedures have been designed to assure that hazards that have the potential to cause personnel injury or destruction of property are identified, managed and/or systematically eliminated from the operation. This system eliminates hazards, limits the potential for injury and increases the overall safety of the work environment.

***Employee Exposure Assessment.*** Employee exposure assessment is performed to identify and evaluate potential exposure hazards associated with the employees work station. The exposure assessment data is used to determine if changes or modifications to the work station are needed to limit exposure to laboratory conditions that could negatively affect an employee's existing medical conditions.

***Bloodborne Pathogens.*** Accutest has implemented the OSHA Bloodborne Pathogen Standard, 29CFR1910.1030 to reduce occupational exposure to Hepatitis B Virus (HBV), Human Immunodeficiency Virus (HIV) and other bloodborne pathogens that employees may encounter in their workplace.

***Respiratory Protection Plan.*** The respiratory protection plan assures that Accutest employees are protected from exposure to respiratory hazards. This program is used in situations where engineering controls and/or safe work practices do not completely control the identified hazards. In these situations, respirators and other protective equipment are used. Supplemental respiratory protection procedures are applied to specified maintenance personnel, employees who handle hazardous wastes in the hazardous waste storage area, and any employee that voluntarily elects to wear a respirator.

***Chemical Hygiene Plan.*** The Chemical Hygiene Plan complies with the requirements of the Occupational Safety and Health Administration's Occupational Exposure to Hazardous Chemicals in the Laboratory Standard, 29 CFR 1910.1450. This plan establishes procedures, identifies safety equipment, personal protective equipment, and work practices that protect employees from the potential health hazards presented by hazardous chemicals in the laboratory if properly used and/or applied.

***Chemical Spill Response Plan.*** The chemical spill response plan has been designed to minimize the risks from a chemical spill or accidental chemical release in the laboratory. Risk minimization is accomplished through a planned response that follows a defined procedure. The staff has been trained to execute spill response procedures according to the specifications of the plan, which identifies the appropriate action to be taken based on the size of the spill.

***Emergency Action & Evacuation Plan.*** The Emergency Action and Evacuation Plan details the procedures used to protect and safeguard ANC's employees and property during emergencies. Emergencies are defined as fires or explosions, gas leaks, building collapse, hazardous material spills, emergencies that immediately threaten life and health, bomb threats and natural disasters such as floods, hurricanes or tornadoes, terrorism or terrorist actions. The plan identifies and assigns responsibility for executing specific roles in situations requiring emergency action. It also describes the building security actions coinciding with the "Alert Condition", designated by the Department of Homeland Security.

***Lockout/Tagout Plan.*** Lockout/tagout procedures have been established to assure that laboratory employees and outside contractors take steps to render equipment inoperable

and/or safe before conducting maintenance activities. The plan details the procedures for conducting maintenance on equipment that has the potential to unexpectedly energize, start up, or release energy or can be operated unexpectedly or accidentally resulting in serious injury to employees. The plan ensures that employees performing maintenance render the equipment safe through lock out or tag out procedures.

***Personal Protection Policy.*** Policies have been implemented which detail the personal protection requirements for employees. The policy includes specifications regarding engineering controls, personal protective equipment (PPE), hazardous waste, chemical exposures, working with chemicals and safe work practices. Safety requirements specific to processes or equipment are reviewed with the department supervisor or the Health and Safety Manager before beginning operations.

***Visitor and Contractor Safety Program.*** A safety brochure is given to all visitors and contractors who visit or conduct business at the facility. The brochure is designed to inform anyone who is not an employee of Accutest Laboratories of the laboratories safety procedures. The brochure directs them to follow all safety programs and plans while on Accutest property. This program also outlines procedures for visitors and contractors in the event of an emergency. Visitors are required to acknowledge receipt and understanding of the Accutest policy annually.

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## Appendix I

### GLOSSARY OF TERMS

**Acceptance Criteria:** specified limits placed on characteristics of an item, process, or service defined in requirement documents.

**Accuracy:** the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

**Analyst:** the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

**Audit:** a systematic evaluation to determine the conformance to quantitative *and qualitative* specifications of some operational function or activity.

**Batch:** environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group.

**Blank:** a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

**Blind Sample:** a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

**Calibration:** to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

**Calibration Curve:** the graphical relationship between the known values, such as concentrations of a series of calibration standards and their instrument response.

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**Calibration Method:** a defined technical procedure for performing a calibration.

**Calibration Standard:** a substance or reference material used to calibrate an instrument. **Certified Reference Material (CRM):** a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation, which is issued by a certifying body.

**Chain of Custody:** an unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples.

**Confirmation:** verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to second column confirmation, alternate wavelength, derivatization, mass spectral, interpretation, alternative detectors or, additional cleanup procedures.

**Corrective Action:** the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

**Data Reduction:** the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

**Demonstration of Capability:** a procedure to establish the ability of the analyst to generate acceptable accuracy.

**Document Control:** the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

**Duplicate Analyses:** the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

**Field of Testing:** NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an updated/improved method are required submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff).

**Laboratory Control Sample (such as laboratory fortified blank, spiked blank, or QC check sample):** a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

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**Matrix:** the component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

**Aqueous:** any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

**Drinking Water:** any aqueous sample that has been designated a potable or potential potable water source. **Saline/Estuarine:** any aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake. **Non-aqueous Liquid:** any organic liquid with <15% settleable solids.

**Solids:** includes soils, sediments, sludges and other matrices with >15% settleable solids.

**Chemical Waste:** a product or by-product of an industrial process that results in a matrix not previously defined.

**Air:** whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

**Biota:** animal or plant tissue, consisting of entire organisms, homogenates, and/or organ or structure specific subsamples.

**Matrix Spike (spiked sample or fortified sample):** a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

**Matrix Spike Duplicate (spiked sample or fortified sample duplicate):** a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

**Method Blank:** a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

**Method Detection Limit:** the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

**National Environmental Laboratory Accreditation Program (NELAP):** the overall National Environmental Laboratory Accreditation Program.

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**NELAC Standards:** the plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference.

**Performance Audit:** the routine comparison of independently obtained *qualitative and quantitative* measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

**Precision:** the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

**Preservation:** refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

**Proficiency Testing:** a means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

**Proficiency Test Sample (PT):** a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

**Quality Assurance:** an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

**Quality Control:** the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

**Quality Manual:** a document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

**Quality System:** a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

**Reporting Limits:** the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user.

**Reagent Blank (method reagent blank or method blank):** a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate

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point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

**Reference Material:** a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

**Reference Method:** a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

**Reference Standard:** a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

**Replicate Analyses:** the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.

**Sample Duplicate:** two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis.

**Spike:** a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

**Standard:** the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

**Traceability:** the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

**Validation:** the process of substantiating specified performance criteria.

**Work Cell:** A defined group of analysts that together perform the method analysis. Members of the group and their specific functions within the work cell must be fully documented. A “work cell” is considered to be all those individuals who see a sample through the complete process of preparation, extraction, or analysis. The entire process is completed by a group of capable individuals; each member of the work cell demonstrates capability for each individual step in the method sequence.

Appendix II : Standard Operational Procedures Directory

# Accutes Northern California - Active SOPs

## ADMINISTRATION

<b>Department: Administration</b>		<b>Effective Date</b>
ADM001-1	Procedure for Conduction Management Reviews	10/5/2009
ADM002-1	Data Security and Integrity Procedure	10/20/2009
ADM003-1	Job Description	10/6/2009
ADM004-0	Procedure for Requisition and Verifying Software	10/26/2009
ADM005-0	Skeleton Form for Initials and Signatures	3/3/2008
ADM007-0	Health & Safety Program EHS HMBP Chemical Hygiene	1/1/2012

## GENERAL CHEMISTRY

<b>Department: General Chemistry</b>		<b>Effective Date</b>
GEN001-3	%Moisture - %Solids SM2540G	7/5/2010
GEN002-2	Acidity SM2310B	3/22/2010
GEN003-3	Alkalinity SM18 2320B	12/27/2010
GEN004-2	Ammonia SM18-4500-NH3 B(Distillation), E(Titration), F(ISE) or G(ISE-Std Additions)	3/16/2010
GEN006-4	Anions by EPA 300.0 / SW9056A	1/4/2011
GEN008-3	Hexavalent Chromium (Cr6+) Water Colorimetric SM19-3500-Cr D	1/6/2011
GEN009-3	Hexavalent Chromium (Cr6+) Soil Colorimetric SW3060A (Digestion) / SW7196A	3/22/2010
GEN010-4	Specific conductance 120.1 / SM2510B	1/27/2012
GEN011-3	COD Chemical Oxygen Demand SM5220D / HACH 8000	6/1/2011
GEN012-4	Cyanide, Total and Amenable: CWA by SM4500-CN-C(Man.Distillation),E (Total),G (Amenable) and RCRA by SW846 9010C (Man. Distillation) and SW846 9014 (Colorimetric & Titration)	2/7/2012
GEN013-2	1664A Oil & Grease (HEM) and TRPH (SGT-HEM)	2/13/2010
GEN014-3	Nitrogen, Combined: Nitrate+Nitrite SM4500-NO3 E Cadmium Reduction	1/6/2011
GEN015-2	Nitrogen, Total Kjeldahl TKN SM4500-NH3-F	3/10/2010
GEN016-2	Nitrogen, Nitrite NO2 as N SM4500-NO2-B	3/15/2010
GEN017-4	Fluoride by ISE SM4500-F-C / SW9214 w/o Distillation	6/1/2011
GEN019-2	Ferrous Iron SM3500-Fe-D Phenanthroline Colormetric Method	4/8/2010

**Uncontrolled Copy**

GEN020-1	pH Hydrogen Ion Concentration 9040B	4/8/2010
GEN021-2	Perchlorate by IC Water and Soil EPA 314.0	2/9/2010
GEN022-2	pH Hydrogen Ion - Soil and Waste 9045C	3/23/2010
GEN023-2	pH Hydrogen Ion Concentration - Water SM4500-H+B	3/16/2010
GEN024-2	Phenolics, Total Recoverable Soil and Water 420.1 / SW9056	3/17/2010
GEN025-3	Total Phosphorus and ortho-Phosphate by SM4500-P E	12/27/2010
GEN026-2	Solids, Settleable (SS) SM2540-F	3/23/2010
GEN027-2	Solids, Total (TS) SM18 2540-B	3/23/2010
GEN028-4	Solids, Total Dissolved (TDS) SM2540-C	1/27/2012
GEN029-4	Solids, Total Suspended (TSS) SM2540-D	4/9/2012
GEN030-2	Sulfide SM4500-S=D / HACH 8131	3/17/2010
GEN031-4	Total Organic Carbon (TOC) SM5310C	5/31/2011
GEN032-2	Turbidity 180.1 / SM2130B	3/15/2010
GEN034-2	Glassware Cleaning for Inorganics and Metals	4/12/2010
GEN036-2	Hexavalent Chromium (Cr6+) Water 7196A	3/24/2010
GEN039-1	Hexavalent Chromium (Cr6+) Water by IC 7199	3/1/2010
GEN040-1	Hexavalent Chromium (Cr6+) Soil by IC 7199	3/1/2010
GEN041-0	Oxidation Reduction Potential Not NELAP	11/15/2009

**METALS AND METALS PREP*****Department: Metals***

		<b><i>Effective Date</i></b>
MET001-3	Mercury in Water by 245.1 or 7470A	3/10/2010
MET002-3	Mercury in Soil 7471A	2/8/2011
MET003-6	Metals by ICP EPA 6010B	2/7/2012
MET004-3	Metals by ICP EPA 200.7	10/8/2010

***Department: Metals Prep***

		<b><i>Effective Date</i></b>
MP001-2	ICP Metals Digestion Aqueous 200.7	2/26/2010
MP002-3	ICP Metals Digestion for Water 3010A	5/1/2011
MP003-2	ICP Metals Digestion Soils 3050B Includes Wipes and Sludge	2/11/2010
MP004-2	STLC Title 22 Waste Extraction Test (WET)	2/10/2012

**ORGANIC PREP, VOLATILES AND SEMI VOLATILES**

**Department: Organic Prep**

**Effective Date**

OP002-4	8081 / 8082 / 608 - Pesticides / PCBs Water Extraction (Liq-Liq Sep Funnel) 3510C	2/10/2012
OP003-1	8082 PCBs Waste Dilution 3580A	12/4/2008
OP004-3	8270 SVOC Soil Extraction (ASE) 3545A and (Waste Dilution) 3580A	10/13/2010
OP005-3	SVOC Water Extraction Liq-Liq Sep Funnel 3510C / 625	2/16/2010
OP006-3	TPH-Extractable_Soil_3545A_ASE	10/12/2010
OP007-2	TPH Water Extraction Liq-Liq Sep Funnel 3510C	2/15/2010
OP008-2	Cleaning Procedure for Organic Glassware	2/23/2010
OP009-2	TCLP Leaching Procedure for Semi-Volatile Organics and Metals	3/2/2010
OP010-0	SPLP Synthetic Leaching Procedure for Semi & Non-Volatile Analytes	4/17/2009
OP011-2	NWTPH-HCID & Dx Extraction for Water	3/5/2010
OP012-3	NWTPH-HCID & Dx Extraction for Soil 3545A (ASE)	11/1/2010
OP013-0	TCLP 1311 Volatile Organics ZHE	10/19/2009
OP014-2	8081A/8082-OC Pesticides & PCBs Soil Extraction by 3550C Sonication	12/20/2011
OP015-0	8270 SVOC Soil Extraction Sonication 3550C	4/28/2010
OP016-0	TPH-Extractable Soil Extraction sonication 3550C	4/28/2010
OP017-0	NWTPH-HCID & Dx Extraction for Soil 3550C Sonication	4/28/2010
OP018-0	8270 SIM PAHs Soil Extraction Sonication 3550C	1/1/2011
OP019-0	Arizona 8015AZR1 Soil Extraction for TPH C10-C32	12/20/2011

**Department: Organic Semi-volatile**

**Effective Date**

SV001-8	8081A / 8082 Organochlorine Pesticides and PCBs Analysis	5/3/2011
SV002-6	Semi-volatile organics (SVOC) by GC/MS 8270C	2/10/2012
SV003-3	Total Petroleum Hydrocarbons-Extractable (TPH) by GC/FID; NWTPH; 8015AZ; AK102; CALUFT; 8015B, 8015D	9/20/2011
SV004-1	NWTPH-HCID analysis	3/8/2010
SV007-0	Semi-volatile organics by GC/MS 625	1/27/2010
SV008-0	608 OC Pesticides & PCBs Analysis	3/11/2010

**Department: Organic Volatile**

**Effective Date**

VO001-8	In Review Volatile Organic Compounds (VOCs) and GRO by GC/MS 8260B	
VO001-7	Volatile Organic Compounds (VOCs) and GRO by GC/MS 8260B	9/20/2011
VO001T1-0	Table: DoD QSMV4.1 8260 Table F-4	1/1/2010
VO002-3	BTEX / MtBE / TPH as Gasoline (GRO) by GC/PID/FID 8021B / 8015B M	9/7/2011
VO003-1	Alcohols by 8015 Direct Aqueous Injection	2/12/2009
VO004-2	Volatile Organics Refrigerator Blanks	5/4/2011
VO005-1	Purge & Trap Soils 5035	5/25/2010
VO006-0	Purge & Trap Waters 5030	2/5/2009
VO008-1	Volatile Organic Compounds (VOCs) by GC/MS 624 CWA	12/6/2010
VO009-0	NWTPH-Gx and VOCs by GC/MS	5/29/2010
VO010-2	NWTPH-Gx by GC/FID	4/4/2011
VO011-0	Soil Sampling for Volatile Organic Compounds for Alaska	9/20/2011

## **PROJECT MANAGEMENT**

### ***Department: Project Management***

### ***Effective Date***

PM001-1	Project Management	10/19/2009
PM001P1-0	Project Management Policy on Client Supplies & Shipping	10/19/2009
PM002-1	Review of Tenders	10/19/2009
PM003-2	Subcontracting	1/4/2010
PM003A-1	Subcontract Report Procedure	10/15/2009
PM004-1	Confidentiality Protection Procedures	9/30/2009

## QUALITY ASSURANCE

**Department: Quality Assurance**

**Effective Date**

QA001-2	Error Correction	7/6/2010
QA002-2	Corrective Action	2/12/2010
QA003-0	Blank Spike Control Charting - database	3/1/2008
QA004-2	Measurement Traceability - Purchasing Services and Laboratory Supplies	2/11/2010
QA005-1	Auto Pipette Verification &/Or Calibration	1/25/2009
QA006-2	MDL / LOD / LOQ Determination	4/7/2010
QA008-9	Quality System Manual for Accutest Laboratories Northern California, Inc. (QSM ALNCa)	8/2/2011
QA009-3	Refrigerator and Freezer Temperatures	3/8/2011
QA010-3	Annual Verification of Thermometer Accuracy	11/29/2010
QA011-2	Test Method Validation, MDL, LOD, LOQ	2/1/2011
QA012-1	Significant Figures	4/14/2009
QA013-2	Manual Integrations	6/17/2009
QA014-3	Personnel Training and Documentation DOC	10/15/2011
QA015-0	Unassigned	
QA016-3	SOP Template with Signatories	8/19/2011
QA017-1	Data Integrity Investigations	10/12/2009
QA018-1	Sample Batching for Prep and Analysis	10/15/2009
QA019-2	Support Equipment and Maintenance	12/10/2010
QA020-3	SOP Preparation, Approval, Distribution and Archiving	8/19/2011
QA021-1	Control of Laboratory Documentation	10/12/2009
QA022-1	Quality Control, Evaluation Criteria (ME and Bias)	10/27/2009
QA023-1	Sample Compositing and Sample Aliquots	10/29/2009
QA024-1	PT Proficiency Testing	4/7/2009
QA025-1	Report Generation and Review	10/6/2009
QA026-0	Sample Homogenization (Representative Solid Sample Aliquot)	1/12/2009
QA027-0	Syringe Calibrations	4/17/2009
QA028-0	Volumetric Dispensers - Critical Volumes	1/20/2009
QA029-1	Volumetric Dispensers - Non-critical Volumes	7/10/2010
QA030-0	Calibration Check for Analytical Balances	2/17/2009
QA031-0	Data Integrity and Ethics Training	10/18/2009
QA032-0	Review of Inorganic Data	10/26/2009
QA033-1	Control of Non-conforming Work	2/21/2011
QA034-0	Quality System Review	9/7/2009
QA035-1	Internal Audits and Preventative Action	2/12/2010
QA036-0	Data Integrity Monitoring	10/23/2009
QA037-0	Data Integrity Issues Reporting	10/20/2009
QA038-1	Client Complaints Resolution	2/21/2011
QA039-0	Control of Records	10/19/2009
QA040-0	In-House QC Criteria - Development and Use	10/25/2009
QA041-0	Method Comparability and Validation	10/26/2009
QA042-0	Review of Organic Data	10/25/2009
QA043-0	Purchasing Services and laboratory Supplies	2/24/2010
QA044-0	Reagent (DI) Water Quality Control	2/24/2010
QA045-1	Use of Calibration Curves	1/27/2012

## **SAMPLE CONTROL**

### ***Department: Sample Control***

### ***Effective Date***

SC001-2	Sample Handling and Login Procedures	1/14/2009
SC002-2	Waste and Sample Disposal	1/5/2009
SC004-1	Sample and Analysis Guide	1/1/2009
SC005-1	Sample Storage	10/23/2009
SC006-0	Sample Containers Quality Control	9/18/2008
SC006-1	Sample Containers Quality Control - Draft	12/8/2011
SC007-0	Bottle Kits; Cooler cleaning, Packaging and Shipping - Draft	10/11/2009
SC008-0	Foreign Soil Handling	12/23/2009
SC009-0	Procedure for Sample Couriers	4/22/2011

## **Appendix III: Analytical Capability**

<b>NELAP No</b>	<b>EPA Method</b>
<b>102 - Inorganic Chemistry of Drinking Water</b>	
102.030.003	EPA 300.0
102.045.001	EPA 314.0
102.220.001	SM4500-NO2 B
102.240.001	SM4500-P E
<b>108 - Inorganic Chemistry of Wastewater</b>	
108.020.001	EPA 120.1
108.381.001	EPA 1664A
108.110.001	EPA 180.1
108.112.001	EPA 200.7
108.120.001	EPA 300.0
108.360.001	EPA 420.1
108.660.001	HACH8000
108.390.001	SM2130B
108.400.001	SM2310B
108.410.001	SM2320B
108.420.001	SM2340B
108.430.001	SM2510B
108.440.001	SM2540B
108.441.001	SM2540C
108.442.001	SM2540D
108.443.001	SM2540F
108.465.001	SM4500-CI G
108.470.001	SM4500-CN C
108.472.001	SM4500-CN E
108.473.001	SM4500-CN G
108.490.001	SM4500-H+ B
108.493.001	SM4500-NH3 D or E (19th/20th)
108.495.001	SM4500-NH3 E (18th)
108.494.001	SM4500-NH3 F or G (18th)
108.510.001	SM4500-NO2 B
108.520.001	SM4500-NO3 E
108.540.001	SM4500-P E
108.580.001	SM4500-S= D
108.611.001	SM5310C

**109 - Toxic Chemical Elements of Wastewater**

109.010.001	EPA 200.7
109.190.001	EPA 245.1
109.808.001	SM3500-Cr B (21st)
109.811.001	SM3500-Cr D (18th/19th)
109.825.001	SM3500-Fe D (18th/19th)

**110 - Volatile Organic Chemistry of Wastewater**

110.040.001	EPA 624
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**111 - Semi-volatile Organic Chemistry of Wastewater**

111.273.001	EPA 1664A
111.170.001	EPA 608
111.100.001	EPA 625
111.272.001	SM5520B (20th)

**114 - Inorganic Chemistry of Hazardous Waste**

114.010.001	EPA 6010B
114.103.001	EPA 7196A
114.106.001	EPA 7199
114.140.001	EPA 7470A
114.141.001	EPA 7471A
114.222.001	EPA 9014
114.240.001	EPA 9040B
114.241.001	EPA 9045C
114.250.001	EPA 9056
114.270.001	EPA 9214

**115 - Extraction Test of Hazardous Waste**

115.030.001	CCR Chapter11, Article 5, Appendix II
115.021.001	EPA 1311
115.040.001	EPA 1312

**116 - Volatile Organic Chemistry of Hazardous Waste**

116.020.009	EPA 8015B
116.040.002	EPA 8021B
116.080.001	EPA 8260B
116.110.001	LUFT
116.100.001	LUFT GC/MS

**117 - Semi-volatile Organic Chemistry of Hazardous Waste**

117.010.001	EPA 8015B
117.210.000	EPA 8081A

117.220.000	EPA 8082
117.110.000	EPA 8270C
117.016.001	LUFT

***120 - Physical Properties of Hazardous Waste***

120.070.001	EPA 9040B
120.080.001	EPA 9045C

### Appendix IV: Equipment List

Equip.	Manufacture& Description	Serial Number	Operating System Software	Data Processing Software	Location
GC-JJ	Agilent 6890 GC / PID-FID Tekmar LSC2000/ G1530A	US00025879	HP Chemstation	HP Chemstation	Organics Volatiles
GC-KK	HP 5890 GC/PID-FID 7673 18593B /18594B / G1205A	3235A46302	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-L	Agilent 6890 GC / 5975C MS / OI 4660A	CN10706098	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-M	Agilent 6890 / 5973N MS Tekmar LSC 2000/G1530A/G1098A	US00021444	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-N	Agilent 6890 GC/ 5973N MS Tekmar LSC 3000/ G1530 A	US00027818	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-V	Agilent 6890 GC/ 5973N MS Tekmar LSC 2000/G2577A/G1098A	US10518041	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-R	Agilent 7890 GC / 5975C MS OI 4551-A	US10452710	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-U	Agilent 7890 GC / 5975C MS OI 4660A	CN11321059	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-Q	Agilent 7890 GC / 5975C MS OI 4551-A	US10452714	HP Chemstation	HP Chemstation	Organics Volatiles
GCMS-W	Agilent 5890 GC / 5975B MS G3172A / 4552 / OI 4660	CN10627031	HP Chemstation	HP Chemstation	Organics Volatiles
GC-FF	HP 5890 GC / FID 767318593A / 18596A / 18594A	3140A38168	HP Chemstation	HP Chemstation	Organics SVOCs
GC-GG	Agilent 68590 GC / FID 767318593B / 18596M / G1512A	US00001432	HP Chemstation	HP Chemstation	Organics SVOCs
GC-HH	Agilent 6890 GC / FID 767318593A / 18596B / G1701BA	3310A48782	HP Chemstation	HP Chemstation	Organics SVOCs
GC-MM	HP 6890/ECD HP 7683 Injector	US00024420	HP Chemstation	HP Chemstation	Organics SVOCs
GC-OO	HP 5890 GC / ECD-ECD 7673 / G1223A / 18593B / 18594B /18596B	3235A44943	HP Chemstation	HP Chemstation	Organics SVOCs
GC-PP	HP 5890 GC / ECD-ECD 7673A G1223A / 18593B / 18594B / 18596B	3115A34621	HP Chemstation	HP Chemstation	Organics SVOCs
GCMS-X	Agilent 5890N GC / 5975B MS 7683B / G3172A / G2913A / G2614A	CN10636038	HP Chemstation	HP Chemstation	Organics SVOCs
GCMS-T	Agilent 6890N GC 5973B MS 7683 Injector 7683 Autosampler	US10123019 US10440808 CN3303176 CN42629441	G1530N G2577A G2613A G2614A	MS Chemstation	Organics SVOCs
GCMS-Y	Agilent 5890N GC / 5975N MS 7683B G3172A / G2613A / G2614A	CN10634077	HP Chemstation	HP Chemstation G1701DA	Organics SVOCs

Equip.	Manufacture& Description	Serial Number	Operating System Software	Data Processing Software	Location
ASE-2	Dionex (Accelerated Solvent Extractor)	07020400	None	None	Organic Prep
ASE-3	Dionex (Accelerated Solvent Extractor)	07030593	None	None	Organic Prep
TUMBLER-1	Tumbler	Bison 508-01-537	None	None	Organic Prep
TUMBLER-2	Tumbler	GTR Tumbler	None	None	Organic Prep
TUMBLER-3	Tumbler	GTR Tumbler	None	None	Organic Prep
Centrifuge	Fisher Scientific	225	None	None	Organic Prep
TV1 TV2 TV3 TV4	Concentrator/ TurboVap LV	TV0626N13155 TV0553N12782 TV9907N18679 TV0105N10106	None	None	Organic Prep
Sonicating Bath	Aquasonic 50T	21811-820	None	None	Organic Prep
Sonicator	Misomix Ultrasonic Liquid Processor	-	None	None	Organic Prep
BAL-1	Adventurer Pro AV8101C	8027331004	None	None	Organic Prep
BAL-2	Sartorius Extend Series ED3202S Has a parallel port	27450089	None	None	Organic Prep
Oven	Oven	1330GZZZZMFG	None	None	Organic Prep
Shaker-1 Shaker-2 Shaker-3 Shaker-4	Shakers	3520 3520 SHKA 2506 SHKA 2506	None	None	Organic Prep

Equip.	Manufacture& Description	Serial Number	Operating System Software	Data Processing Software	Location
VORTEX-1	Vortex	Genie2	None	None	Organic Prep
VORTEX-2	Vortex	Genie2	None	None	Organic Prep
Mercury Ur	Mercury Amalgam Unit	Amalgam System	None	None	Mercury
BLOCK-1	Block Digester-ICP	SC100 SCP Digiprep 24	None	None	Metals Prep
BLOCK-2	Block Digester-ICP	SC100 SCP Digiprep 24	None	None	Metals Prep
BLOCK-3	Block Digester-ICP	SC100 SCP Digiprep 36	None	None	Metals Prep
BLOCK-4	Block Digester-Hg	05-C0530 Digiprep 24	None	None	Metals Prep
FIMS-1	Mercury Analyzer/ FIMS-100	1522	None	None	Metals Prep
LEEMAN	Teledyne-Leeman	Hydra II	None	None	Metals Prep
ICP-MS	Agilent 7500a	JP10300382	None	None	Metals
ICP-2	Thermo Fisher Scientific iCAP6000	ICP-20104815	None	None	Metals
MET-1	Balance XL-500	SN08675	None	None	Metals Prep
IC-1	Dionex IC1100/AS-40	12060164 (IC) AS-40(Autosampler)	Chromeleon	Chromeleon	Inorganics
IC-2	Dionex ICS2000/AS40/AS16G	04030440 (IC) 09010526 (Autosampler)	Chromeleon	Chromeleon	Inorganics
IC-3	Dionex ICS3000/ASDV/NG1	08120173 (Det) 08050056 (Pump) 09101340 (Autosampler) 07080459 (VIS Lamp)	Chromeleon	Chromeleon	Inorganics
BLOCK-5	Digester – TKN – Labconco # 23012 Controller for Digester (230V 15.96Amp)	080587801E 081195750E			Inorganics
Meter-1	Meter, ISE	090756	None	None	Inorganics
Meter-2	Meter, ISE	008802	None	None	Inorganics
Meter-3	pH Meter	001103	None	None	Inorganics

Equip.	Manufacture& Description	Serial Number	Operating System Software	Data Processing Software	Location
Meter-4	Meter, ISE	B19733	None	None	Inorganics
Vacuum Pump	Vacuum Pump	DOA-P704-AA	None	None	Inorganics
Chiller	1170	305421	None	None	Inorganics
Chiller	1171MD	F09900116	None	None	Inorganics
Meter	Conductivity 162A	017809	None	None	Inorganics
COD	COD Reactor 16500-10	2329	None	None	Inorganics
Vacuum Pump	Vacuum Pump	DOA-P704-AA	None	None	Inorganics
Vacuum Pump	Vacuum Pump	400-1901	None	None	Inorganics
SPE	Manifold-6 position	-	None	None	Inorganics
Centrifuge	Sorvall Legend XT	40970026	None	None	Inorganics
Hot Plate	PC-600D	013606254903	None	None	Inorganics
Hot Plate	Hot Plate	980061119531	None	None	Inorganics
NTU-1	Nephelometer 2100AN	05090C020055	None	None	Inorganics
OVEN-1	Oven 1350GM	0200403	None	None	Inorganics
OVEN-2	Oven OV702G	2039090215821	None	None	Inorganics
SPEC-1	Spectrophotometer DR/2500 Odyssey	011000001384	None	None	Inorganics
SPEC-2	Spectrophotometer Genesys 20	35GL317012	None	None	Inorganics
TOC	TOC Autosampler/Analyzer Phoenix 8000	US06209002	None	None	Inorganics
WCL-1	Balance AG204	1119362894	None	None	Inorganics
WCL-2	Balance BA-610	20701774	None	None	Inorganics



**APPENDIX B**  
**EPA/ADEQ POLICIES AND PROCEDURES**

## **Arizona Data Qualifiers**

**(12/11/2000)**

**(Developed by the Sub-committee of the Arizona Environmental Laboratory Advisory Committee)**

### **Microbiology:**

- A1 = Too numerous to count.
- A2 = Sample incubation period exceeded method requirement.
- A3 = Sample incubation period was shorter than method requirement.
- A4 = Target organism detected in associated method blank.
- A5 = Incubator/water bath temperature was outside method requirements.
- A6 = Target organism not detected in associated positive control.
- A7 = Micro sample received without adequate headspace.

### **Method blank:**

- B1 = Target analyte detected in method blank at or above the method reporting limit.
- B2 = Non-target analyte detected in method blank and sample, producing interference.
- B3 = Target analyte detected in calibration blank at or above the method reporting limit.
- B4 = Target analyte detected in blank at/above method acceptance criteria.

### **Confirmation:**

- C1 = Confirmatory analysis not performed as required by the method.
- C2 = Confirmatory analysis not performed. Confirmation of analyte presence established by site historical data.
- C3 = Qualitative confirmation performed. See case narrative.
- C4 = Confirmatory analysis was past holding time.
- C5 = Confirmatory analysis was past holding time. Original result not confirmed.

### **Dilution:**

- D1 = Sample required dilution due to matrix interference. See case narrative.
- D2 = Sample required dilution due to high concentration of target analyte.
- D3 = Sample dilution required due to insufficient sample.
- D4 = Minimum reporting level (MRL) adjusted to reflect sample amount received and analyzed.

### **Estimated concentration:**

- E1 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not possible due to insufficient sample.
- E2 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not performed due to sample matrix.
- E3 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not performed due to holding time requirements.

- E4 = Concentration estimated. Analyte was detected below laboratory minimum reporting level (MRL).
- E5 = Concentration estimated. Analyte was detected below laboratory minimum reporting level (MRL), but not confirmed by alternate analysis.
- E6 = Concentration estimated. Internal standard recoveries did not meet method acceptance criteria.
- E7 = Concentration estimated. Internal standard recoveries did not meet laboratory acceptance criteria.

**Hold time:**

- H1 = Sample analysis performed past holding time. See case narrative.
- H2 = Initial analysis within holding time. Reanalysis for the required dilution was past holding time.
- H3 = Sample was received and analyzed past holding time.
- H4 = Sample was extracted past required extraction holding time, but analyzed within analysis holding time. See case narrative.

**BOD:**

- K1 = The sample dilutions set-up for the BOD analysis did not meet the oxygen depletion criteria of at least 2 mg/L. Any reported result is an estimated value.
- K2 = The sample dilutions set up for the BOD analysis did not meet the criteria of a residual dissolved oxygen of at least 1 mg/L. Any reported result is an estimated value.
- K3 = The seed depletion was outside the method acceptance limits.
- K4 = The seed depletion was outside the method and laboratory acceptance limits. The reported result is an estimated value.
- K5 = The dilution water D.O. depletion was > 0.2 mg/L.
- K6 = Glucose/glutamic acid BOD was below method acceptance criteria.
- K7 = A discrepancy between the BOD and COD results has been verified by reanalysis of the sample for COD.

**Laboratory fortified blank/blank spike:**

- L1 = The associated blank spike recovery was above laboratory acceptance limits. See case narrative.
- L2 = The associated blank spike recovery was below laboratory acceptance limits. See case narrative.
- L3 = The associated blank spike recovery was above method acceptance limits. See case narrative.
- L4 = The associated blank spike recovery was below method acceptance limits. See case narrative.

Note: The L1, L2, L3 & L4 footnotes need to be added to all corresponding analytes for a sample.

**Matrix spike:**

- M1 = Matrix spike recovery was high, the method control sample recovery was acceptable.
- M2 = Matrix spike recovery was low, the method control sample recovery was acceptable.
- M3 = The accuracy of the spike recovery value is reduced since the analyte concentration in the sample is disproportionate to spike level. The method control sample recovery was acceptable.
- M4 = The analysis of the spiked sample required a dilution such that the spike concentration was diluted below the reporting limit. The method control sample recovery was acceptable.

M5 = Analyte concentration was determined by the method of standard addition (MSA).

**General:**

N1 = See case narrative.

N2 = See corrective action report.

**Sample quality:**

Q1 = Sample integrity was not maintained. See case narrative.

Q2 = Sample received with head space.

Q3 = Sample received with improper chemical preservation.

Q4 = Sample received and analyzed without chemical preservation.

Q5 = Sample received with inadequate chemical preservation, but preserved by the laboratory.

Q6 = Sample was received above recommended temperature.

Q7 = Sample inadequately dechlorinated.

Q8 = Insufficient sample received to meet method QC requirements. QC requirements satisfy ADEQ policies 0154 and 0155.

Q9 = Insufficient sample received to meet method QC requirements.

Q10 = Sample received in inappropriate sample container.

Q11 = Sample is heterogeneous. Sample homogeneity could not be readily achieved using routine laboratory practices.

**Duplicates:**

R1 = RPD exceeded the method control limit. See case narrative.

R2 = RPD exceeded the laboratory control limit. See case narrative.

R3 = Sample RPD between the primary and confirmatory analysis exceeded 40%. Per EPA Method 8000B, the higher value was reported.

R4 = MS/MSD RPD exceeded the method control limit. Recovery met acceptance criteria.

R5 = MS/MSD RPD exceeded the laboratory control limit. Recovery met acceptance criteria.

R6 = LFB/LFBD RPD exceeded the method control limit. Recovery met acceptance criteria.

R7 = LFB/LFBD RPD exceeded the laboratory control limit. Recovery met acceptance criteria.

R8 = Sample RPD exceeded the method control limit.

R9 = Sample RPD exceeded the laboratory control limit.

**Surrogate:**

S1 = Surrogate recovery was above laboratory acceptance limits, but within method acceptance limits.

S2 = Surrogate recovery was above laboratory and method acceptance limits.

S3 = Surrogate recovery was above laboratory acceptance limits, but within method acceptance limits. No target analytes were detected in the sample.

S4 = Surrogate recovery was above laboratory and method acceptance limits. No target analytes were detected in the sample.

S5=Surrogate recovery was below laboratory acceptance limits, but within method acceptance limits.

S6 = Surrogate recovery was below laboratory and method acceptance limits. Reextraction and/or reanalysis confirms low recovery caused by matrix effect.

S7 = Surrogate recovery was below laboratory and method acceptance limits. Unable to confirm matrix effect.

S8 = The analysis of the sample required a dilution such that the surrogate concentration was diluted below the method acceptance criteria. The method control sample recovery was acceptable.

S9 = The analysis of the sample required a dilution such that the surrogate concentration was diluted below the laboratory acceptance criteria. The method control sample recovery was acceptable.

S10 = Surrogate recovery was above laboratory and method acceptance limits. See Case narrative.

**Method/analyte discrepancies:**

T1 = Method promulgated by EPA, but not by ADHS at this time.

T2 = Cited ADHS licensed method does not contain this analyte as part of method compound list.

T3 = Method not promulgated either by EPA or ADHS.

T4 = Tentatively identified compound. Concentration is estimated and based on the closest internal standard.

**Calibration verification:**

V1 = CCV recovery was above method acceptance limits. This target analyte was not detected in the sample.

V2 = CCV recovery was above method acceptance limits. This target analyte was detected in the sample. The sample could not be reanalyzed due to insufficient sample.

V3 = CCV recovery was above method acceptance limits. This target analyte was detected in the sample, but the sample was not reanalyzed. See case narrative.

V4 = CCV recovery was below method acceptance limits. The sample could not be reanalyzed due to insufficient sample.

V5 = CCV recovery after a group of samples was above acceptance limits. This target analyte was not detected in the sample. Acceptable per EPA Method 8000B.

**0155.000**                    **ANALYTICAL METHODS HAVING PROVISIONS FOR A ONE-  
POINT CALIBRATION AND CONTINUING CALIBRATION  
VERIFICATION CONSTRAINTS POLICY**

**Level One**                    **Arizona Department of Environmental Quality**

**Originator:**                Kenyon C. Carlson, Manager  
Quality Assurance\Quality Control(QA\QC) Unit

**Contact For  
Information:**                Kenyon C. Carlson, Manager  
Quality Assurance\Quality Control(QA\QC) Unit

**Issue Date:**                October 23, 1998

**PURPOSE**

Most analytical methods have established upper and lower control limits for CCV's and when the recovery exceeds those limits the method is considered out-of-control. ADEQ is concerned with the assumption that the 'data are not impacted', as reported by laboratories when the upper control limit of a CCV has been exceeded in a non-detect result. Currently, there is no way to differentiate between an instrument that has gained sensitivity and one that has drifted out of control when the upper control limit of a CCV is ignored.

Adherence to this policy will assure that all laboratory-generated data submitted to ADEQ meets regulatory requirements and are legally defensible.

Because ADEQ is a regulatory agency, compliance results must be able to meet all legal requirements. Where CCV requirements are part of the test method and where test methods are part of the regulatory requirements, then the CCV requirements as dictated by the analytical method must be followed.

**AUTHORITY**

A.A.C. R18-4-106 and R9-14-608.

The EPA methods continue to be written such that upper and lower control limits for the CCV are established and there is no documentation which permits one to ignore the violation of an upper control limit in light of certain conditions.

## **DEFINITIONS**

**Continuing Calibration Verification Standard (CCV)**--Consists of an aliquot of reagent water to which known quantities of the method analytes are added by the laboratory. The CCV's purpose is to determine whether the methodology is in control by verifying the linearity of the calibration curve and to assure that the sample results reflect accurate and precise measurements.

**Data**--For the purposes of this policy, data is defined as raw data (examples include but are not limited to calibration curves, chromatograms, spectras, injection logs, etc.) and does not include laboratory reports. (Contact the QA unit for further information).

## **POLICY**

From a regulator's perspective, a laboratory must follow the method as written to ensure the analytical data generated is defensible and can survive the scrutiny of litigation. ADEQ will not accept test results for regulatory purposes when the CCV's acceptance criteria have been exceeded. This includes sample results where the upper control limit of the CCV has been exceeded and the result is reported as non-detect.

However, in the event a CCV exceeds its control limits for a detect sample, ADEQ allows the laboratory to either 1)recalibrate the entire multi-point curve and reanalyze the samples or 2) perform a one-point calibration as the method permits.

## **RESPONSIBILITY**

The ADEQ QA/QC staff will be responsible, when reviewing data for the purpose of recommending to ADEQ program staff to either accept or reject such data, to ensure that the procedures outlined in this policy are followed.

## **APPLICABILITY**

This policy is only applicable to those methods which provide for a one-point calibration and those water matrices for the analysis of volatile organic compounds (VOCs), synthetic organic compounds (SOCs), and inorganic compounds (IOCs) analyzed using 40 CFR methods (ex. 200, 500, and 600 series). This policy does not apply to those samples analyzed using SW-846 methods.

## **LABORATORY PROCEDURES**

EPA and the ADEQ QA/QC Unit require that laboratories which elect to recalibrate using a one-point calibration must demonstrate there is adequate instrument sensitivity to detect a peak at the

method reporting level for those contaminants. Therefore, to justify reporting sample results as non-detect when the control limits of a CCV have been exceeded, the laboratory must recalibrate using a standard at the method reporting level and re-run all the samples or extracts after that CCV.

The laboratory must detect a significant peak for each analyte reported in the method reporting level standard. A significant peak is considered to be one in which the peak is at least 3 to 5 times the signal to noise ratio (40 CFR, Part 136, Appendix B, Procedure section 1a).

This ADEQ policy provides a means for laboratories to demonstrate that sample results are, in fact, non-detect for target analytes. The method reporting level standard must be analyzed (and determined to be acceptable) before reanalyzing any samples in a run.

#### Non-detects:

To report a non-detect result using a one-point calibration, the laboratory must meet the following requirement: Establish the absence of a significant peak at the retention time of the target analyte. The absence of a significant peak at the retention time of the target analyte is defined as one whose response is less than that of the analyte present in the low level standard (which must be prepared at the reporting limit) used for the one-point calibration.

#### Detects:

To report a detect result using a one-point calibration, a laboratory must meet the following requirement: a one-point calibration must be performed so that the concentration of the one-point calibration standard is within  $\pm 20\%$  of the concentration of analyte detected in a sample.

## ATTACHMENT

### STATEMENT OF POSITION

There has been some debate among the laboratory community concerning continuing calibration verification (CCV's) standards and non detect samples. Most analytical methods have established upper and lower control limits for CCV's and when the recovery exceeds those limits the method is considered out of control. Recently, there has been a growing consensus among some laboratories that an analytical method is *not* out of control if the upper control limit of the CCV is exceeded providing the sample is a non-detect. The reasoning here is that the instrument has somehow "gained" sensitivity and if there were anything in the sample, it would surely have been detected.

The ADEQ QA/QC Unit understands this logic and recognizes that it may true in some cases. However, this is only one of several possibilities. Another possibility is that the analytical method is now out of control. ADEQ is concerned with the assumption that the 'data are not impacted', as reported by laboratories when the upper control limit of a CCV has been exceeded in a non-detect result. Currently, there is no way to differentiate between an instrument that has gained sensitivity and one that has drifted out of control when the upper control limit of a CCV is ignored.

As a regulatory agency, ADEQ cannot assume that each time the upper control limit is exceeded, it is the result of increased instrument sensitivity. Such an assumption can result in the court or the hearing officer invalidating or dismissing the analytical results because an integral portion of the method's quality control has been omitted. The ADEQ Quality Assurance\Quality Control Unit has discussed this subject at length with EPA Region IX's Quality Assurance Management Section. Region IX concurs with the ADEQ's QA\QC Unit's interpretation. They have further expressed their concern that ignoring established upper control limits for the CCV is not in line with "good laboratory science" and may invite abuse and even laboratory fraud.

**APPENDIX C**

**URS DATA VERIFICATION PROCEDURES**

**Contractor Project No.:** \_\_\_\_\_

**Laboratory Report No.:** \_\_\_\_\_

**Completeness Check By:** \_\_\_\_\_

**Date:** \_\_\_\_\_

### DATA REVIEW: COMPLETENESS CHECK

Review 100% of data when the Contractor first receives the final laboratory report (1) to verify that all laboratory analyses that were requested were performed and reported and (2) to perform a cursory quality control review to identify if any laboratory test results need further work or reanalysis. The screening check will included the following:

<b>Completed</b>	<b>Screening Check</b>
	Are all analyses that are requested on the Chain-of-Custody and any change orders present in the data package?
	Does the data package include a copy of the Chain-of-Custody forms?
	Has the laboratory placed any data qualifier flags on the analytical results?
	Does the laboratory's case narrative identify problems, including an explanation of flagged data?
	Does the data package include reports for:
	a. Method and trip blanks?
	b. Matrix spikes?
	c. Matrix spike duplicates and/or sample duplicates?
	d. A blank spike/laboratory control sample/second source check sample?
	e. Surrogates?
	Based on any missing information and/or gross quality control exceedences, should the laboratory perform additional analytical work on the samples before holding times have expired or the leftover sample is discarded? (Data collected to characterize investigation-derived waste will not be further reviewed by a contractor QA Officer unless there is a specific concern about the quality of the data or unless requested by the ADEQ Project Manager.)

**Contractor Project No.:** \_\_\_\_\_  
**Laboratory Report No.:** \_\_\_\_\_  
**Completeness Check By:** \_\_\_\_\_  
**Date:** \_\_\_\_\_

**DATA REVIEW: DATA VERIFICATION**

Perform data verification on all samples collected to characterize the site, including quarterly groundwater monitoring samples and soil investigation samples. Data verification will be performed by a chemist or other professional with data validation or analytical laboratory experience who is approved by ADEQ. The professional should be familiar with the QC requirements specified for the analytical methods being reviewed. Data verification precedes data validation and is a systematic process for evaluating whether data has been generated with acceptable quality control, as defined in the Project QAPP.

Review only the items listed below, as well as completeness of supporting documentation. This is a cursory review of the laboratory's quality control and may suggest that a more thorough validation is needed.

Completed	Review Item
	Case Narrative Have any anomalies, deficiencies, and QC problems been identified in the case narrative? What corrective action, if any, was taken?
	<b>Chain-of-Custody Documentation</b> Are the original Chain-of-Custody forms with ID numbers and laboratory receipt signatures present?
	Are there copies of internal tracking documents, as applicable?
	<b>Sample Analysis Results</b> Are sample analysis results included for environmental samples, with quantitation limits (include dilutions and reanalyses)?
	<b>QC Summary</b> Is the following information included?
	Initial and continuing calibrations
	Method blanks, continuing calibration blanks, and preparation blanks
	Surrogate percent recoveries
	Internal standard percent recoveries
	Matrix spike percent recoveries
	Laboratory duplicate relative percent differences
	Laboratory QC check sample, laboratory control sample recoveries
	Field duplicates, if identified, reproducibility will be evaluated
	Acceptance criteria, if not already established by the method/DQO
	Definitions for any laboratory data qualifiers used
	Method of standard additions (INORGANIC)
	ICP serial dilution (INORGANIC)
	<b>Specifically review the following:</b> Was a check for timeliness and errors conducted, including requested deliverables, preservation, holding times, and Chain-of-Custody?
	Was a duplicate sample/matrix spike/matrix spike duplicate/postdigest spike reviewed

**Contractor Project No.:** \_\_\_\_\_

**Laboratory Report No.:** \_\_\_\_\_

**Completeness Check By:** \_\_\_\_\_

**Date:** \_\_\_\_\_

<b>Completed</b>	<b>Review Item</b>
	against precision and accuracy criteria specified by the method or by project DQOs?
	Were compound quantitation and reported detection limits reviewed, checking reporting limits against contract required limits, verifying dry weights, calculations, and dilutions?
	<b>Does the Verification Report include the following information?:</b> Case narrative including, but not limited to, an overall summary of data acceptability and comparison to DQOs and DQIs (PARCC), a list of recommended changes, a summary of all laboratory contacts in which communications with the laboratory, if any, would be identified, and any other problems associated with the actual analysis which might impact the sample integrity or data quality
	Marking of recommended changes directly on copies of the laboratory reports for the client's ease in performing data entry
	Tabulated summary of all data results supplied electronically by email or on 3.5-inch floppy disks in a commonly used software format

**Contractor Project No.:** \_\_\_\_\_  
**Laboratory Report No.:** \_\_\_\_\_  
**Completeness Check By:** \_\_\_\_\_  
**Date:** \_\_\_\_\_

**DATA REVIEW: DATA VALIDATION**

Experienced chemists will perform full data validation on a data package(s) selected by the contractor Project Manager at the beginning of the project. The package(s) should be a full sample batch (approximately 20 samples), consisting of samples collected for groundwater monitoring and/or soil investigation, and should be typical of the type of samples expected for the project. Each analytical method used in the project should be initially validated prior to proceeding with performing data verification on the bulk of the laboratory results. Additionally, during each six-month period that the project is ongoing, the Project Manager will select additional data packages for validation which are representative of the matrix and analyses being performed.

Data validation will consist of a review of sample and QC results, and the accompanying raw data. The ADEQ Project Manager will identify the compounds of concern, and the data validation will include a review of 100% of the QC data and sample data for these compounds in the laboratory report for a sample delivery group. Compounds not identified as contaminants of interest will not be validated unless requested by ADEQ's Project Manager. Data validation will be conducted by the contractor's QA officer or an independent data validation contractor. The ADEQ QA Unit will validate data at the ADEQ Project Manager's request. Validation includes all of the following items listed as validation deliverables.

The percentage of data that undergoes full validation may be increased if substantial data quality issues are raised during the initial or subsequent assessments. Or, ADEQ may require that a larger percent of the data be fully validated for various reasons including, but not limited to, determining the extent of the issue and/or if the issue has been corrected in subsequent analyses, or that additional data be made available for review, besides the validation deliverables mentioned below.

Completed	Review Item
	<b>Case Narrative</b> Have any anomalies, deficiencies, and QC problems been identified in the case narrative? What corrective action, if any, was taken?
	<b>Chain-of-Custody Documentation</b> Are the original Chain-of-Custody forms with ID numbers and laboratory receipt signatures present?
	Are there copies of internal tracking documents, as applicable?
	<b>Sample Analysis Results</b> Are sample analysis results included for environmental samples, with quantitation limits (include dilutions and reanalyses)?
	<b>QC Summary</b> Is the following information included? Initial and continuing calibrations
	Method blanks, continuing calibration blanks, and preparation blanks
	Surrogate percent recoveries
	Internal standard percent recoveries

**Contractor Project No.:** \_\_\_\_\_  
**Laboratory Report No.:** \_\_\_\_\_  
**Completeness Check By:** \_\_\_\_\_  
**Date:** \_\_\_\_\_

Completed	Review Item
	Matrix spike percent recoveries
	Laboratory duplicate relative percent differences
	Laboratory QC check sample, laboratory control sample recoveries
	Field duplicates, if identified, reproducibility will be evaluated
	Acceptance criteria, if not already established by the method/DQO
	Definitions for any laboratory data qualifiers used
	Gas chromatograph breakdown products
	Retention times and acceptance windows (ORGANIC)
	ICP interference check sample (INORGANIC)
	Method of standard additions (INORGANIC)
	ICP serial dilution (INORGANIC)
	<b>Raw data, chromatograms, and area quantitation reports (ORGANIC), sequential measurement readout records for ICP, graphite furnace atomic absorption (AA), flame AA, cold vapor mercury, cyanide, and/or other inorganic analyses (INORGANIC), including but not limited to the following:</b> Environmental samples (include dilutions and reanalyses)
	Instrument tuning, for analyses of gas chromatography/mass spectrometry (GC/MS)
	Initial calibration and continuing calibrations
	Method blanks, continuing calibration, and preparation blanks
	Surrogate recoveries and internal standard recoveries, where applicable
	Matrix spike (MS)
	Laboratory duplicate or matrix spike duplicate (MSD)
	Laboratory QC check sample, or laboratory control samples, as applicable
	Retention time windows
	Percent moisture for soil samples
	Sample extraction and cleanup logs (ORGANIC)
	Enhanced spectra of target analytes and tentatively identified compounds (TICs) with the associated best match spectra for MS data
	Sample digestion and/or sample preparation logs (INORGANIC)
	Instrument analysis log for each instrument used (INORGANIC)
	Postdigest spikes (INORGANIC)
	Method of standard additions when applicable (INORGANIC)
	ICP serial dilution (INORGANIC)
	Instrument tuning for ICP/MS, when applicable (INORGANIC)
	<b>Specifically review the following:</b> Was a check for timeliness and errors conducted, including requested deliverables, preservation, holding times, and Chain-of-Custody?
	Was a duplicate sample/matrix spike/matrix spike duplicate/post-digest spike reviewed

**Contractor Project No.:** \_\_\_\_\_

**Laboratory Report No.:** \_\_\_\_\_

**Completeness Check By:** \_\_\_\_\_

**Date:** \_\_\_\_\_

<b>Completed</b>	<b>Review Item</b>
	against precision and accuracy criteria specified by the method or by project DQOs?
	Was compound quantitation and reported detection limits reviewed, checking reporting limits against contract required limits, verifying dry weights, calculations, and dilutions?
	Was target list compounds identified, indicating proper identification of analytes?
	Was sample result verification conducted, in which the final reports are reviewed against all raw instrumental data and logs and all applicable worksheets to check anomalies, data reduction/calculations, transcription, linear ranges, and dilutions?
	<b>OPTIONAL (as requested by ADEQ for data validation on a case-by-case basis)</b>
	Method detection limits (MDLs)
	Instrument detection limits (IDLs)
	ICP linear range (INORGANIC)
	<b>Does the Validation Report include the following information?:</b> Case narrative including, but not limited to, an overall summary of data acceptability and comparison to DQOs (PARCC), a list of recommended changes, a summary of all laboratory contacts, in which communications with the laboratory, if any, would be identified, and any other problems associated with the actual analysis which might impact the sample integrity or data quality
	Marking of recommended changes directly on copies of the laboratory reports for the client's ease in performing data entry
	Tabulated summary of all data results supplied electronically by email or on 3.5-inch floppy disks in a commonly used software format