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Subject: Work Plan for Soil Sampling  
Lead Impacted Soil near Potable Water Tank at Devils Postpile National Monument  
Madera County, California

Dear Mr. Winters:

Environmental Cost Management, Inc. (ECM) has prepared this Work Plan for conducting additional site characterization near the potable water tank (Site) at Devils Postpile National Monument (DEPO) (**Figure 1**). Because high concentrations of lead exist in soil at the site, the National Park Service (NPS) is addressing their remediation under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) as a non-time-critical removal action (NTCRA). As part of this process, ECM is preparing an Engineering Evaluation and Cost Analysis (EE/CA) to assist the NPS with assessing several removal action alternatives and identifying the preferred alternative. ECM will obtain new investigation data to estimate the area and volume of impacts to soil and identify removal action alternatives.

This work plan discusses the purpose, rationale and proposed sampling locations and analysis of potentially lead-impacted soil. The details of sample collection, laboratory analysis, and quality assurance activities are included in the attached sampling and analysis plan (SAP) and quality assurance project plan (QAPP). ECM field personnel will conduct all work under the attached health and safety plan (HASP) created specifically for this project and the DEPO site.

## 1 BACKGROUND

In 2008, Provost and Pritchard Consulting Group (P&P)<sup>1</sup> conducted a preliminary assessment (PA) of a historical release of lead-based paint chips and sandblasting debris at the 100,000-gallon aboveground potable water tank at DEPO. The tank site is located on a slope northeast of the campground (**Figure 2**). NPS has reviewed all available site information and concluded that the PA did not completely characterize the nature and extent of lead contamination at the Site. ECM reviewed the data from the report and will perform sampling using incremental sampling methodology (ISM) to facilitate the completion of the EE/CA for the site.

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<sup>1</sup> Provost and Pritchard Engineering Group, Inc. *Preliminary Assessment for the National Park Service, Devils Postpile National Monument*, prepared for Sequoia and Kings Canyon National Park, Three Rivers, California. October 2008.

## 1.1 SITE DESCRIPTION

Located on the western slope of the Sierra Nevada range between 7,200 and 8,200 feet (**Figure 1**), DEPO contains an interesting assemblage of flora, fauna and geology, for which the monument was set aside. The highlight of the monument is a sheer wall of symmetrical basaltic columns more than 60 feet high. The formation is a remnant of a basalt flow worn smooth on top by glacial action.

DEPO is located along the Middle Fork of the San Joaquin River Valley, which drops more than 100 feet at Rainbow Falls, located two miles by trail from the Devils Postpile formation. DEPO covers approximately 798 acres administered under the auspices of the NPS. The Ansel Adams Wilderness encompasses over 93 percent of DEPO<sup>2</sup>, and the monument provides a portal to High Sierra backcountry.

DEPO is located in extreme northeastern Madera County and closely borders Mono County, California. The monument is located approximately two miles southwest of Mammoth Mountain ski resort at 37.629 N Longitude and 119.0847 Latitude. DEPO features a ranger station, a 21-site campground, and eight miles of established trails.

Access to DEPO from Lee Vining is 25 miles on Highway 395 to State Route 203, the Mammoth Lakes turnoff. Drive 3.7 miles through the town of Mammoth Lakes to a junction with Minaret Summit Road, which forks to the right toward the Devils Postpile National Monument. Take the right fork and drive about 5.6 miles to the monument entrance<sup>3</sup>. From June to September a shuttle bus from the Mammoth Visitor Center to the Mammoth Mountain Ski Area parking lot conveys visitors over Minaret Summit and down to the monument<sup>4</sup>.

The closest community to DEPO is Mammoth Lakes, located nine miles to the east in Mono County, California (**Figure 1**). According to the 2010 Census, the town of Mammoth Lakes had a total population of 8,234. Other nearby communities along U.S. Highway 395 include Bishop, Lee Vining, Crestview, and Toms Place.

Two Native American (American Indian) lands are located within Madera County, the North Fork Rancheria and the Picayune Rancheria. The Big Sandy Rancheria, Cold Springs Rancheria and the Table Mountain Rancheria are located in adjacent Fresno County.

Nine archeological sites were recorded during a post-fire survey of the entire monument<sup>5</sup>. Previous work in the park includes a reconnaissance survey and a clearance survey<sup>6</sup>. Two of the sites date to the historic period. The others are chipped stone scatters. The small number of diagnostic artifacts recovered from the surveys that have been conducted at DEPO date from 3,000 B.C. to A.D. 500. A gap in diagnostic artifacts from the period A.D. 500 – A.D. 1500 coincides with a presumed hiatus in occupation due to major volcanic activity in the region.

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<sup>2</sup> National Park Service, "Devils Postpile Park Statistics", last updated 05/26/2013, accessed 05/29/2013, <http://www.nps.gov/depo/parkmgmt/statistics.htm>

<sup>3</sup> Grossi, Mark. "Devils Postpile National Monument" Longstreet Highroad Guide to the California Sierra Nevada. Sherpa Guides, ([http://www.sherpaguides.com/california/mountains/eastern\\_sierra/devils\\_postpile\\_national\\_monument.html](http://www.sherpaguides.com/california/mountains/eastern_sierra/devils_postpile_national_monument.html))

<sup>4</sup> National Park Service, "Devils Postpile Reds Meadow and Devils Postpile Shuttle Information", last updated 05/29/2013, accessed 05/29/2013, <http://home.nps.gov/depo/planyourvisit/gettingaround.htm>

<sup>5</sup> Hull, K. L., Hale, M. R. *Post-fire Archaeological Survey of Devils Postpile National Monument, Madera County, California*. Dames & Moore, Inc., Chico, California.

<sup>6</sup> Wells, Susan J., *An Archeological Survey Plan for the Western Region of the National Park Service*, Western Archeological and Conservation Center, National Park Service, Department of the Interior, 1994.

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### 1.1.1 SITE GEOLOGY AND GEOHYDROLOGY

DEPO is located high on the western slope of the Sierra Nevada in eastern California. The Sierra Nevada is the largest single mountain range in the contiguous United States and is bounded on the west by California's Central Valley and on the east by the Basin and Range Province. Physiographically, the Sierra Nevada is a section of the Cascade-Sierra Mountains province, which in turn is part of the larger Pacific Mountain System physiographic division. The core of this north-northwest trending range is an enormous intrusion of granitic rock, the Sierra Nevada batholith<sup>7</sup>.

#### Geology/Geologic History

DEPO lies within a geological transition zone. The western half of the monument consists of 85-million-year-old granitic bedrock of the Sierra Nevada batholith, while the eastern half reflects the Quaternary age volcanics of the eastern Sierra.

According to a United States Geologic Survey (USGS) summary of the geologic history of the area,<sup>8</sup> this region is part of the Long Valley Caldera, which erupted explosively about 750,000 years ago. The oldest volcanic rock in the vicinity of the Monument is believed to be the basalt now exposed at The Buttresses, although its exact age is not known. The valley of the Middle Fork of the San Joaquin River was glaciated more than once prior to about 760,000 years ago. Deposition of the tuff near Reds Meadow about 760,000 years ago was concurrent with extensive volcanic activity that created the Long Valley caldera and produced the Bishop Tuff<sup>9</sup>. Stream erosion, perhaps aided by another glaciation, removed much of the tuff from the central part of the valley. Rhyodacite lava erupted from vents just downstream from the present site of the Postpile and flowed southward beyond Rainbow Falls. Andesite lava erupted near Mammoth Pass and cascaded into the Middle Fork valley. Less than 100,000 years ago basalt, now exposed at the Devils Postpile, erupted north of Pumice Flat in the area of Soda Springs and flowed out to cover several square miles of the valley floor. The lava flowed down the valley like a river until it was blocked by a natural dam created by a glacial moraine left down-valley by a receding glacier during a previous ice age. The lava began filling the valley behind this dam, creating a lava lake 400 up to feet deep. As the lava flow ceased, the molten rock began cooling slowly from the top down. Contraction stresses developed and jointing occurred, forming the columns. Stream and glacial erosion again removed much of the accumulated volcanic rock from the Middle Fork valley. The last glaciation, which ended about 10,000 years ago, produced the polish and striations visible on the top of the Devils Postpile.

The Red Cones, and their associated lava flow, were formed sometime after the last glacier vanished from the valley. Pumice erupted from the Inyo and Mono Craters and covers the area as a surface deposit. This area is still volcanically active and closely monitored by the USGS.

#### Soils

Soil quality and productivity depend on climate, inherent soil type, and soil condition. Soils within the project area are predominantly derived from granite. Soil depths range from less than

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<sup>7</sup> U.S.G.S., "Geology in the Parks – Geology and Geophysics - Sierra Nevada", last updated 01/13/04, accessed 05/29/2013. <http://geomaps.wr.usgs.gov/parks/province/pacifmt.html>.

<sup>8</sup> U.S.G.S., "Devils Postpile National Park Geologic Story", Adapted from pamphlet "Geologic Story of Devils Postpile" by N. King Huber, USGS, and Wymond W. Eckhardt, NPS, last updated 09/07/2000, accessed 05/29/2013, <http://www.nature.nps.gov/geology/usgsnps/depo/dpgeol1.html>

<sup>9</sup> Geological Survey Professional Paper 554—D, *Tuff of Red Meadow, Cenozoic Volcanic Rocks of the Devils Postpile Quadrangle, Eastern Sierra Nevada California*, 2007.

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1 inch to feet<sup>10</sup>. High elevation restricts the growing season and maintains cold soil temperatures for most of the year in all but the southern, lower elevation sites. This limits the activity of plants, burrowing animals, soil insects, and microorganisms. Essential plant nutrients, such as nitrogen, phosphorus, potassium, calcium, and magnesium are severely limited<sup>11</sup>.

Other sources of parent material include areas of volcanics, including andesite, basalt, and rhyolite, and pyroclastic deposits. Andesitic tuffs, ash, and pumice soils were observed near the tank during the ECM's site reconnaissance. Most of the high elevation meadows are rich in volcanic ash. Soils formed in tephra and ash tend to be richer in nutrients and organic matter, but when exposed can also be exceptionally dusty.

Meadow soils are derived from alluvial deposits and glacial debris. They tend to be very deep, well stratified, and relatively free of rock fragments and rich in decaying organic matter. Most wet and moist meadows have seasonally high water tables, primarily in spring and early summer. Meadow soils that are often saturated are more susceptible to rutting and compaction, which reduces infiltration and can lead to gully erosion.

### Hydrology

The Madera subbasin of the San Joaquin Valley Groundwater Basin in California consists of lands overlying the alluvium in Madera County<sup>12</sup>. The subbasin is bounded on the south by the San Joaquin River, on the west by the eastern boundary of the Columbia Canal Service area, on the north by the south boundary of the Chowchilla Subbasin, and on the east by the crystalline bedrock of the Sierra Nevada foothills. Major streams in the area include the San Joaquin and Fresno Rivers. Average annual precipitation is 11 inches throughout the majority of the subbasin and 15 inches in the Sierra foothills.

The San Joaquin River transforms throughout Devils Postpile, from a broad, low-gradient meander to scattered pools, fast-flowing rapids, cascades, and finally culminating at Rainbow Falls, a waterfall that stands 101 feet tall. The Devils Postpile Wetland Inventory and Condition Assessment<sup>13</sup> revealed that 8.5% of the Monument is wetlands,

Changes in groundwater levels are based on annual water level measurements by DWR and cooperators. Water level changes were evaluated by quarter township and computed through a custom Department of Water Resources (DWR) geostatistics computer program. On average, the subbasin water level has declined nearly 40 feet from 1970 through 2000. The period from 1970 through 1978 showed steep declines totaling about 30 feet. The nine-year period from 1978 to 1987 saw stabilization and rebound of about 25 feet, taking the water levels close to where they were in 1970. The period 1987 through 1996 again showed steep declines, bottoming out in 1996 at about 45 feet below 1970 levels. Water levels rose about eight feet from 1996 to 2000. Water levels declines have been more severe in the eastern portion of the subbasin from 1980 to the present, but the western subbasin showed the strongest declines before this time.

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<sup>10</sup> Provost and Pritchard Engineering Group, Inc., *Preliminary Assessment for the National Park Service, Devils Postpile National Monument*, prepared for Sequoia and Kings Canyon National Park, Three Rivers, California. October 2008.

<sup>11</sup> USDA, Forest Service, *Final Wilderness Plan and Environmental Impact Statement, Inyo and Sierra National Forests, John Muir/Ansel Adams and Dinkey Lakes*, July, 2012.

<sup>12</sup> Department of Water Resources, *California's Groundwater*, Bulletin 118, Update 2003.

<sup>13</sup> National Park Service Water Resources Division, *Devils Postpile National Monument Wetland Inventory and Condition Assessment*, April 2008.

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### 1.1.2 CLIMATE, VEGETATION, AND WILDLIFE

Climate within DEPO varies greatly by season. Daytime temperatures can range from the mid-70s to mid-80s degrees Fahrenheit (°F). Evening temperatures can drop into the low 40s (and even the low 30s in the months of September and October). Precipitation usually occurs year round with sub-tropical thunderstorms in the spring and fall and significant rain and snow events in the winter. Average rainfall is about 30 inches. Snowfall exceeds 400-inches per year<sup>14</sup>.

The Inyo National Forest surrounds Devils Postpile National Monument on three sides. Three-quarters of the Monument is included within the Ansel Adams Wilderness. Devils Postpile National Monument's vegetation is a montane forest dominated by red fir and lodgepole pine of the east slope of the Sierra Nevada. Though technically a west slope location, the monument's proximity to both west and east sides of the Sierra Nevada results in biological communities that have east-slope as well as west-slope affinities<sup>15</sup>. Western slope flora includes mountain hemlock, red fir, alder, and gooseberry.

Recent plant inventories documented over 400 plant species in the monument. Along the San Joaquin River and the few creeks that flow into it, typical montane riparian vegetation can be found, such as quaking aspen, black cottonwood, alder, and willows. Both wet and dry meadows dot the monument, and during the spring and early summer when water is available, wildflowers such as cinquefoil and alpine shooting star can be found.

The unique geography of the area fosters relatively high species diversity concentrated in a small area. The Monument contains animals such as black bears, mule deer, and coyotes. Soda Springs Meadow, near the Ranger Station, harbors an abundance of songbirds. Dark-eyed juncos and white-crowned sparrows are common in the summer. The talus at the base of Devils Postpile is home to many squirrels and chipmunks and the pine martens, which hunt them. Another asset in terms of biodiversity is the burned area near Rainbow Falls, which is habitat for many plants and animals that will not live in heavily forested areas.

A total of 135 plant species in the Sierra Nevada have status as Threatened, Endangered, or Sensitive. Plants that are Federal species of concern (former Category 2 species) under the Federal Endangered Species Act include:

1. Three-bracted Onion,
2. Yosemite Woolly Sunflower,
3. Congdon's Lomatium,
4. Tiehm's Rock-cress,
5. Slender-stemmed Monkeyflower, and
6. Bolander's Clover.

Although Category 2 was abolished in 1996, species of concern is an informal term that refers to those species that might be declining or be in need of concentrated conservation actions to prevent decline. Therefore, these six species continue to be evaluated and managed by the National Park Service.

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<sup>14</sup> National Park Service, "Devils Postpile Plan Your Visit", last updated 05/25/2013, accessed 05/29/2013, <http://www.nps.gov/depo/naturescience>

<sup>15</sup> National Park Service, "Devils Postpile Nature & Science", last updated 05/25/2013, accessed 05/29/2013, <http://www.nps.gov/depo/naturescience>

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Four state-listed rare plant species are considered restricted and limited throughout all or a significant portion of their range, and may represent disjunct populations at the extreme end of their range:

1. Yosemite Onion,
2. Tompkin's Sedge,
3. Congdon's Woolly Sunflower, and
4. Congdon's Lewisia.

Endangered or threatened species of animals that occur in the Sierra Nevada include:

1. Sierra Nevada Bighorn Sheep
2. California Condor
3. Southwestern Willow Flycatcher
4. Paiute Cutthroat Trout
5. Lahontan Cutthroat Trout
6. Owens Tui chub

Sierra Nevada mid and high elevations provide the only habitat for the Sierra Nevada mountain yellow-legged frog, the Yosemite toad, and the Sierra Nevada bighorn sheep

### 1.1.3 LAND USES

DEPO hosted 87,845 visitors in 2012, with an average of 103,258<sup>16</sup> visitors annually from 2009 to 2012<sup>17</sup>. Recreational activities vary with the season and include wildflower and wildlife viewing, sightseeing and photography, hiking, cycling, restricted mountain biking, horseback riding, camping, fishing, skiing, and snowshoeing<sup>18</sup>. Approximately 94% of DEPO is wilderness (747 acres).

## 1.2 SITE HISTORY

The Devils Postpile feature was known locally in the 1890s as the Devils Woodpile. It was first recognized as the Devils Postpile in 1901 on various maps. The Postpile was part of Yosemite National Park in the late 1800s when Congress designated its boundaries. Congress removed 500 square miles, including DEPO, from Yosemite National Park in 1905 under pressure from mining and lumber lobbying interests. By 1910, a proposal was made to dynamite the Postpile and use it dam the San Joaquin River. Members of the Sierra Club and University of California professor Joseph LeConte, who was also a mountaineer, successfully campaigned against the project.<sup>19</sup>

On July 6, 1911, President William Howard Taft proclaimed the area a national monument and extended full protection of the federal government to the Devils Postpile formation and Rainbow Falls. The monument was originally administered by the United States Department of

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<sup>16</sup> National Park Service, "Devils Postpile Park Statistics", last updated 05/26/2013, accessed 05/29/2013, <http://www.nps.gov/depo/parkmgmt/statistics.htm>

<sup>17</sup> *Ibid.*

<sup>18</sup> National Park Service, "Devils Postpile Outdoor Activities", last updated 05/26/2013, accessed 05/29/2013, <http://www.nps.gov/depo/planyourvisit/outdooractivities.htm>

<sup>19</sup> Sherpa Guides, Grossi, Mark, "Longstreet Highroad Guide to the California Sierra Nevada – Devils Postpile National Monument", [http://www.sherpaguides.com/california/mountains/eastern\\_sierra/devils\\_postpile\\_national\\_monument.html](http://www.sherpaguides.com/california/mountains/eastern_sierra/devils_postpile_national_monument.html).

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Agriculture (USDA) Forest Service (USFS), and then transferred to the national park system in 1934. After the transfer, DEPO was managed first by Yosemite and then by Sequoia and Kings Canyon National Parks before becoming an independent unit of the national park system. Congress also included 747 acres of the monument in the Ansel Adams Wilderness in 1984; consequently, over 90 percent of the monument is designated as Wilderness.

The NPS oversees the 798-acre Devils Postpile National Monument, while the USFS manages the lands surrounding the monument. Together these two federal agencies work as partners to manage public lands in this area. In 2009, the USFS and the NPS entered into a Memorandum of Understanding (MOU) to collaborate on the preparation of the Devils Postpile National Monument General Management Plan (GMP) and create a foundation for future cooperation in management and planning. Under the MOU, the USFS and DEPO are key participants in the development of desired valley-wide conditions for facilities, transportation, and the overall visitor experience, as well as resource management issues<sup>20</sup>.

### 1.2.1 WATER TANK OPERATIONAL HISTORY

The lead-impacted soils addressed in this project surround an aboveground steel tank that is the sole potable water storage facility for DEPO<sup>21</sup>. The 100,000-gallon tank was installed prior to 1940 and has been in seasonal use since its installation. The tank is drained at the end of each season in late October or early November, and is treated and refilled at the beginning of each spring season in approximately late April or early May. The tank sits on a slope northeast of the campground in the northern part of the monument. The campgrounds, trails, ranger station, and facilities are located within 1,000 feet west and 125 feet below the tank site (**Figure 2**). The site is accessible to NPS employees via a walking trail uphill (to the east) from the campgrounds and the unpaved access road from the east. The site is generally not visible or accessible to visitors.

By 2005, the tank's outer surface had weathered to the point that the paint was peeling and flaking. In September 2005, a painting contractor (AA-1 Services of Paramount, California) was retained to sandblast and recoat the exterior of the water tank. The contractor constructed a 5-foot negative pressure containment system by wrapping scaffolding surrounding the tank with a plastic material. Penetrations were made in the containment for air and vacuum. After operations were complete, however, the NPS supervisor, Mr. John Fernandes, noted that lead-based paint chips and sandblasting material had been left outside the tank and accessible to the public and wildlife. The paint chips and blast material were not removed within 24 hours as required by contract, but remained on the ground for two weeks.

### 1.3 SUMMARY OF PREVIOUS INVESTIGATIONS

In November 2005, DEPO collected soil samples at ten locations within the containment area to verify the cleanup procedures of the painting contractor. Due to a contract violation in which the contractor collected confirmation samples without oversight by NPS staff, soil samples were collected by Mr. Fernandes. The exact locations from which the soil samples were collected are not known; however, notes included in the file and discussions with the supervisory ranger suggest that the samples were collected from within the footprint of the containment area at

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<sup>20</sup> National Park Service, Devils Postpile National Monument General Management Plan. Preliminary Alternatives. Newsletter #3, Summer 2011.

<sup>21</sup> Provost and Pritchard Engineering Group, Inc., *Preliminary Assessment for the National Park Service, Devils Postpile National Monument*, prepared for Sequoia and Kings Canyon National Park, Three Rivers, California. October 2008.

approximately 15-foot centers<sup>22</sup>.

The laboratory analytical results indicated that lead was present in each of the samples above the method reporting limit of 5.0 milligrams per kilograms (mg/kg). The average concentration is approximately 1,049 mg/kg. The maximum lead concentration detected was 2,100 mg/kg and the minimum concentration was 20 mg/kg.

In 2008, at the request of the NPS, Provost and Pritchard Consulting Group (P&P) conducted a Preliminary Assessment (PA) in general accordance with the CERCLA guidance manual for the 2005 release of lead-based paint chips and sand-blasting debris, at the 100,000-gallon aboveground potable water tank at DEPO. The objective of the PA was to identify past and present practices related to the historic release and evaluate the site's Hazard Ranking System score (HRS).

The scope of the investigation included review of available records, a site reconnaissance and interviews with DEPO personnel. The investigation focused on the 2005 water tank sandblasting operations activities intended to remove the lead-based paint from the exterior of the tank.

The PA resulted in the following findings:

- The primary type of waste generated on site was a one-time release of lead-based paint chips related to the sandblasting operations for external tank cleaning in preparation recoating. It is also likely that some amount of the blasting materials were also released to the soil during the blasting operations in 2005. However, the sandblasting material is not considered an environmental hazard.
- Laboratory results for the 10 samples showed that:
  - Lead was present in all 10 samples above the method detection limit.
  - The average lead concentration was approximately 1,049 mg/kg; sample-specific lead concentrations ranged from 20 mg/kg to 2,100 mg/kg. These concentrations of lead in site soils are below the California Human Health Screening Levels (CHHLS) of 3,500 mg/kg for commercial/industrial use.
  - The average lead concentrations slightly exceed the Total Threshold Limit Concentration (TTLC) of 1,000 mg/kg, as defined in Title 22, California Code of Regulations.
  - The concentrations also exceed the EPA regional screening levels (RSLs) for soil (400 mg/kg [residential] and 800 mg/kg [industrial]).
- Based on the PA, groundwater and surface water targets are not within sufficient distance of the tank site for there to be a migratory pathway to these resources. Restrictive air flow due to the hilly forested terrain between the source and potential targets make it unlikely that an airborne pathway exists. However, if soils were to be excavated in the future, the quantity of hazardous substances should be identified.
- The exceedances of soil screening levels for lead indicate that additional information was necessary to determine background concentrations and, if appropriate, to develop proposed action levels for the Site.

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<sup>22</sup> Provost and Pritchard Engineering Group, Inc. Preliminary Assessment for the National Park Service, Devils Postpile National Monument, prepared for Sequoia and Kings Canyon National Park, Three Rivers, California. October 2008.

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## 2 PURPOSE

As summarized above, the 2008 assessment indicated lead, a CERCLA hazardous substance, was present at elevated concentrations in the surface soils around the potable water tank. Because the locations of the samples collected are unknown, the extent of contamination is unknown, which represents a gap in the characterization.

NPS reviewed all available Site information and concluded that the PA did not completely characterize the nature and extent of contamination for purposes of conducting a Non-Time-Critical Removal Action (NTCRA). NPS determined that a non-time-critical removal action was required to address the known and potential threats to public health, welfare, and the environment at the site. To address gaps in the characterization of contamination and to develop and evaluate removal action alternatives in accordance with CERCLA, NPS issued an EE/CA Approval Memorandum on October 11, 2012.

ECM will collect additional site data to characterize the nature and extent of potential lead contamination in soil near the potable water tank at DEPO during the execution of the Work Plan. ECM will evaluate the data to determine whether concentrations of lead exceed site-specific action levels and are therefore subject to a response action. If a removal action is required, the EE/CA will provide a comparative analysis of alternatives for clean up or removal of the hazardous substance based on protectiveness to human health and the environment and implementation cost.

NPS needs the following information to optimize the removal action process:

- The quantity (volume and area) of lead-impacted soil exceeding cleanup goals;
- The extent of lead impacts above background; and
- Quantify the number of paint chip fragments as a function of distance from the water tank.

ECM planned an investigation in accordance with Interstate Technology and Regulatory Council (ITRC) guidance<sup>23,24</sup> to provide information that will allow ECM to recommend a compliant, cost-effective removal action alternative.

### 2.1 QUANTITY OF LEAD

A review of investigation data<sup>25</sup> indicates that lead concentrations exceeding USEPA screening criteria or the RCRA regulatory level for hazardous waste were present at select sampling locations at the site, but the extent of such impacts is uncertain. No sample locations are available for some of the historical samples.

### 2.2 BACKGROUND CONCENTRATION

A clear understanding of the background lead concentrations in soil is important because concentrations of chemicals of concern below the naturally occurring background levels are not generally subject to the removal action under CERCLA<sup>26</sup>.

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<sup>23</sup> ITRC, Technical/Regulatory Guidelines, *Characterization and Remediation of Soils at Closed Small Arms Firing Ranges*, January 2003.

<sup>24</sup> ITRC, Technical and Regulatory Guidance, *Incremental Sampling Methodology*, February 2012.

<sup>25</sup> Provost and Pritchard Engineering Group, Inc., *Preliminary Assessment for the National Park Service, Devils Postpile National Monument*, prepared for Sequoia and Kings Canyon National Park, Three Rivers, California. October 2008.

<sup>26</sup> U.S. EPA, *Role of Background in the CERCLA Cleanup Program*, OSWER 9285.6-07P, April 26, 2002.

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## 2.3 SURFACE PARTICLE SURVEY

ECM will survey of the number of visible lead particles at the surface, per square foot. Estimates will be obtained from the center of the target area, as well as at the edge of the target area, and outside the target area. NPS will use this information to assess potential condor lead particle ingestion, as well as quantify lead particles for remedial soil screening, if warranted.

## 3 FIELD SAMPLING

ECM will implement the incremental sampling methodology<sup>27</sup> (ISM) to characterize the naturally occurring background lead concentrations and the nature and extent of lead contamination at the site. The ISM is a technique designed to statistically reduce or limit variability associated with discrete sampling. It provides a more representative and reproducible estimate of the mean concentration of analytes in a specific area of interest, known as a *decision unit* (DU). ECM anticipates sampling to include collecting ISM samples from the following approximate decision units at the site (**Figure 3**):

1. Surface soils within 5 feet of the water tank, approximately 298 cubic feet (ft<sup>3</sup>)
2. Surface soils between 5 feet and 15 feet away from the water tank, approximately 848 ft<sup>3</sup>
3. Surface soils between 15 feet and 25 feet away from the water tank, approximately 1,940 ft<sup>3</sup>
4. Background soils, approximately 1,000 ft<sup>3</sup>

ECM will conduct fieldwork in two stages: 1) DU boundary and grid demarcation and 2) soil sample collection. ECM may modify the DU extents during the first stage of fieldwork. DU's size and shape may be adjusted in the field depending upon observed paint-chip count and distribution.

ECM will use ISM to collect four samples, each consisting of 30 increments (subsamples), to characterize each decision unit at the site. ECM will collect a total of four MI samples per decision unit. Therefore ECM will collect a total of 120 sample increments to complete four MI samples per decision unit, for a total of 480 approximately 50 gram (g) sample increments during the field activities. The sampling method will be such that the total amount of soil collected in the field from the 30 subsamples for each MI sample will be at least 1 kg of soil depending on the grain size of the sample material. NPS-required QA/QC samples will also be collected.

The SAP further describes the details of the ISM sampling for each site, and the QAPP describes QA/QC samples.

## 3.1 WASTE DISPOSAL

ECM does not anticipate generating waste during this investigation. However, any small quantities of wash and rinse water from decontamination of small equipment will be containerized in a 5-gallon bucket or other container as appropriate and relinquished to DEPO for proper disposal. Field personnel will collect all soil samples from the surface using disposable gloves and/or scoops and place the samples in appropriate containers for transport to the laboratory.

## 4 REPORTING

The EE/CA will present data from this investigation, as well as the previously collected data from the PA report, to assess appropriate remedial efforts for the sites. ECM will prepare the

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<sup>27</sup> ITRC, Technical and Regulatory Guidance, *Incremental Sampling Methodology*, February 2012.

EE/CA in accordance with the USEPA's *Guidance on Conducting Non-Time-Critical Removal Actions under CERCLA*<sup>28</sup>. The EE/CA will establish removal action objectives, document the identified ARARs, analyze cost-effective removal alternatives, assess risk, and recommend a preferred removal alternative that best meets the removal objectives.

ECM anticipates the EE/CA may include some combination of the following alternatives, depending on the results of this investigation:

- No Action;
- Institutional Controls (ICs);
- *In-situ* treatments; and/or
- Excavation and off-site disposal.

The EE/CA will optimize the various alternatives to meet ARARs in the most cost-effective manner.

Best regards,

**ENVIRONMENTAL COST MANAGEMENT, INC.**

  
Holly A. Trejo, P.G.  
Project Manager/Senior Geologist

Cc: Steve Mitchell, NPS  
Gary Kramer, NPS

Figures:

Figure 1: Site Location Map

Figure 2: Site Features

Figure 3: Decision Unit Layout

Attachment A: Sampling and Analysis Plan (with Quality Assurance Project Plan)

Attachment B: Health and Safety Plan

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<sup>28</sup> U.S. EPA, Office of Solid Waste and Emergency Response, EPA/540-R-93-057, *Guidance on Conducting Non-Time-Critical Removal Actions Under CERCLA*, Publication 9360.0-32, August 1993.

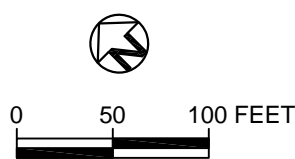
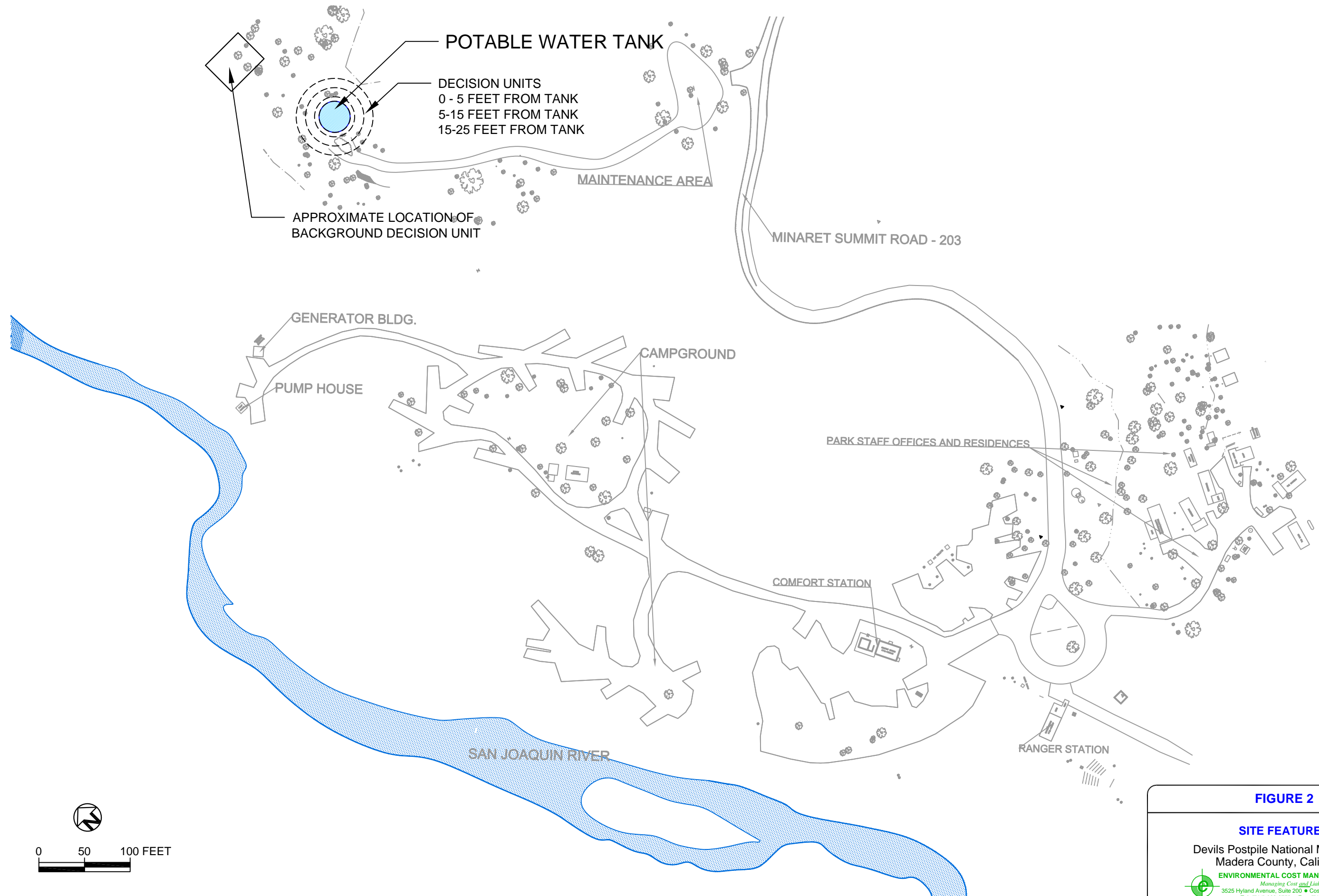
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## Figures









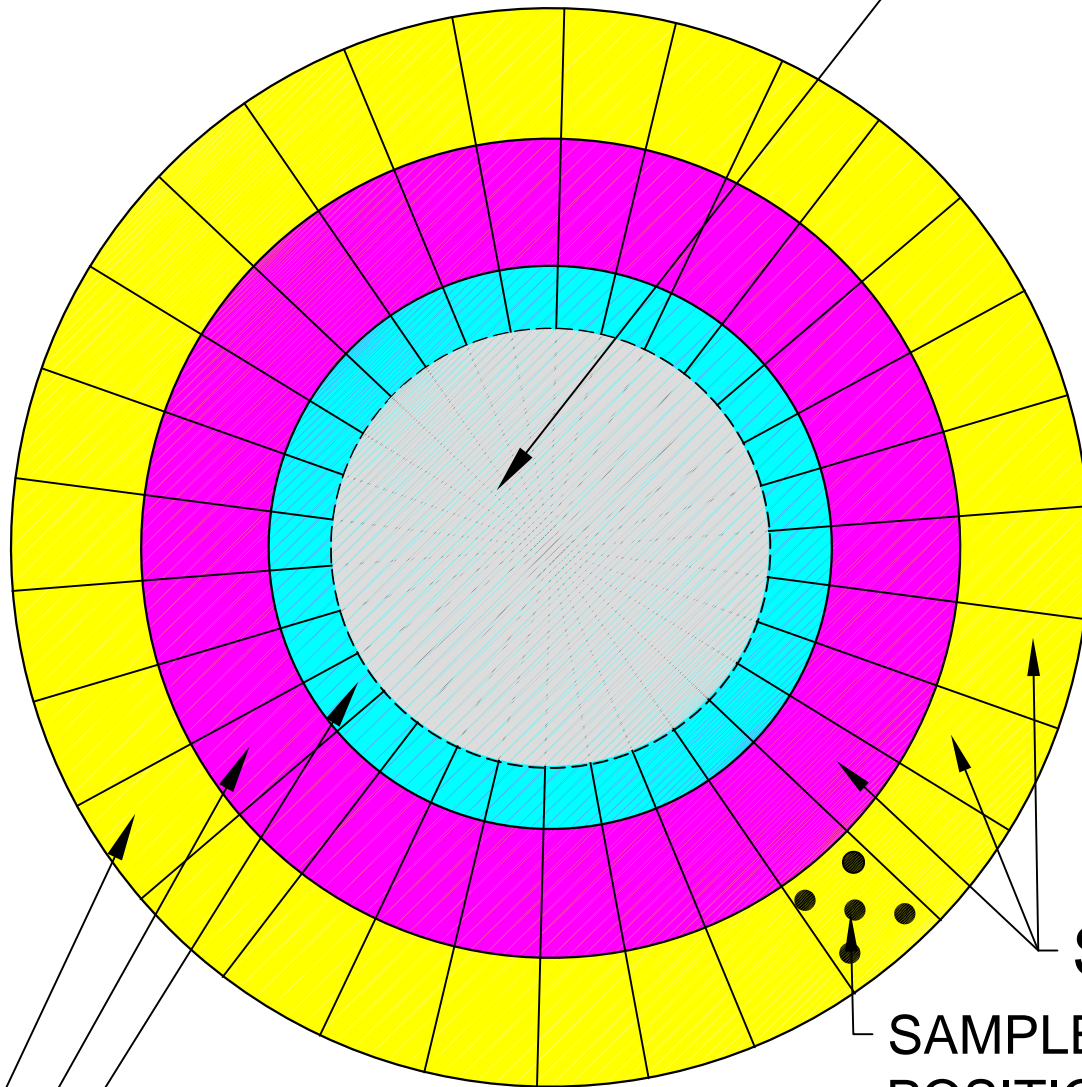
**FIGURE 2**

**SITE FEATURES**

Devils Postpile National Monument  
Madera County, California

**ENVIRONMENTAL COST MANAGEMENT, INC.**  
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# POTABLE WATER TANK



SUs

SAMPLE  
POSITIONS

DU 1: 0 - 5 FEET FROM TANK

DU 2: 5 - 15 FEET FROM TANK

DU 3: 15 - 25 FEET FROM TANK

DU = Decision Unit - 4 samples  
SU = Sample Unit - 30 SUs per DU  
SAMPLE POSITIONS - 5 per SU to be selected  
randomly



0 1,000 15 FEET

**FIGURE 3**

## DECISION UNIT LAYOUT

Devils Postpile National Monument  
Madera County, California



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**Attachment A: Sampling and Analysis Plan and  
Quality Assurance Project Plan**

Prepared for:

U.S. Department of the Interior  
National Park Service  
401 West Hillcrest Drive  
Thousand Oaks, CA 91360

**Sampling and Analysis Plan and  
Quality Assurance Project Plan**  
for  
Site Characterization near Potable Water Tank  
Devils Postpile National Monument  
Madera County, California

June 17, 2013

Prepared By:



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June 17, 2013  
Date

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Holly Trejo, P.G.  
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June 17, 2013  
Date



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Figure 1: Site Location Map (see Work Plan)

Figure 2: Project Organization

Figure 3: Preliminary Conceptual Site Model

## **APPENDICES**

Appendix A: Field Sampling Forms

Appendix B: Laboratory Quality Assurance Manual (LQM)

## ACRONYMS AND ABBREVIATIONS

COC	Chain of custody
COPC	Chemical of potential concern
CQC	Contractor quality control
DEPO	Devils Postpile National Monument
DQO	Data quality objective
DU	Decision Unit
EE/CA	Engineering Evaluation and Cost Analysis
ECM	Environmental Cost Management, Inc.
EPA	United States Environmental Protection Agency
°F	Degree Fahrenheit
GPS	Global positioning system
HASP	Health and safety plan
HDPE	High density polyethylene
IDW	Investigation-derived waste
ISM	Incremental Sampling Methodology
ITRC	Interstate Technology and Regulatory Council
LCS / LCSD	Laboratory control sample / Laboratory control sample duplicate
LPM	Laboratory Project Manager
LQM	Laboratory Quality (Assurance) Manual
MCLs	Maximum contaminant levels
MDL	Method Detection Limit
MQO	Measurement quality objective
MS/MSD	Matrix spike/ matrix spike duplicate
MI	<i>MULTI INCREMENT</i> ® (sampling, see ISM)
NPS	U.S. Department of the Interior, National Park Service
No.	Number
PA	Preliminary Assessment
PARCC	Precision, accuracy, representativeness, completeness, and comparability
PM	Project Manager
PCB	Polychlorinated Biphenyl
PPE	Personal protective equipment
PQL	Practical Quantitation Limit
PRG	Preliminary remediation goal
P&P	Provost and Pritchard Consulting Group
QA	Quality Assurance
QA/QC	Quality assurance/quality control
QAM	Laboratory Quality Assurance Manager
QAPP	Quality assurance project plan
QCM	ECM Quality Control Manager



SAP	Sampling and analysis plan
SOP	Standard operating procedure
SOW	Statement of Work
TPH	Total Petroleum Hydrocarbon
TTLC	Total Threshold Limit Concentration
USACE	U.S. Army Corps of Engineers
yd <sup>3</sup>	Cubic yard

# 1 INTRODUCTION

Environmental Cost Management, Inc. (ECM) is pleased to submit this *Sampling and Analysis Plan and Quality Assurance Project Plan* (SAP/QAPP) to the United States Department of the Interior, National Park Service (NPS), for the additional characterization of soil near the potable water tank in Devils Postpile National Monument (DEPO) in Madera County, California (**Figure 1** of Work Plan). Soil characterization will be used to complete an Engineering Evaluation and Cost Analysis (EE/CA). This document consists of two parts: the Sampling and Analysis Plan (SAP), and the Quality Assurance Project Plan (QAPP).

Previous work has characterized the soil with concentrations of lead above the Total Threshold Limit Concentration (TTLC) of 1,000 mg/kg, as defined in Title 22, California Code of Regulations. Exceedances of TTLC's for lead indicate that additional information is necessary to determine background concentrations and, if appropriate, to develop proposed action levels (PALs) for the Site. The characterization information is necessary to evaluate remedial options if it is determined that lead-impacted soils pose a risk to human health or to the environment.

ECM prepared this SAP/QAPP for NPS under Contract No. P12PX12152.

# 2 PROJECT ORGANIZATION AND RESPONSIBILITY

The project quality control (QC) organization ensures the data meet the project objectives and the specific requirements outlined in the SAP/QAPP. A description of the roles of the project personnel responsible for overall implementation of the project is presented below. **Figure 2** depicts this structure as an organization chart.

## 2.1.1 Project Manager, NPS – DEPO

The **NPS - DEPO Project Manager**, Jonathan Winters, has overall responsibility for the project. Mr. Winters serves as the Contracting Officer's Representative for purposes of ensuring that ECM meets the technical requirements of their contract with NPS.

Specific responsibilities of the NPS project manager include:

- Provide technical direction to ECM during all phases of the project;
- Plan and coordinate meetings and communications between the various parties involved in the project;
- Review, comment, and approve the SAP and EE/CA report; and
- Coordinate NPS oversight of field activities.

## 2.1.2 Program Manager, ECM

The **ECM Program Manager**, Andrew Campbell, PE, is responsible for coordinating the ECM effort and communication with NPS on status of the project. Specific responsibilities of the ECM Program Manager include the following:

- Approval of the draft and final Work Plan and the SAP/QAPP;
- Communication with NPS on a monthly basis to provide an update on the project status;
- Coordinating ECM activities to meet NPS project goals and timeline; and
- Review of the draft and final Engineering Evaluation/Cost Analysis (EE/CA) Report.

### 2.1.3 Project Manager, ECM

The ECM **Project Manager** (PM), Holly Trejo, is in charge of day-to-day management in support of DEPO project. She is responsible for the overall sampling event. Additional PM responsibilities include:

- Writing the SAP/QAPP, Work Plan, and HASP; making sure the plans are followed during field investigation;
- Interacting with the laboratory to ensure samples are received, sample condition is verified, the correct analysis is conducted, and assuring laboratory results are provided in a timely manner. Examples include:
  - Developing and maintaining a sample-tracking matrix based on the chains-of-custody;
  - Using the chains-of-custody to monitor sampling events to assure data quality;
  - Using the chains-of-custody to document the collection of quality assurance (QA) and QC samples;
  - Using the chains-of-custody to document any deviation from the SAP and to document variances to the SAP; and
  - Using the chains-of-custody to alert the laboratory of any sampling problems in order that corrective actions can be implemented to ensure that laboratory analytical data is as complete and of acceptable quality
- Interacting with field personnel to correct any out-of-control field conditions that may affect the quality of analytical data, including coordinating re-sampling or other field corrective action;
- Interacting with the ECM QA/QC Manager and the laboratory to address out-of control situations that may require re-sampling or re-analysis, and
- Interacting with ECM QA/QC manager and laboratory project manager to verify that data meets the project data quality objectives (DQOs).

The PM or PM's designated Project Chemist will be responsible for the production of summary tables of all analytes measured above detection limits as well as presenting qualified, assessed, and verified data. Additionally, the PM or Project Chemist will prepare a summary text detailing the analytical results and any anomalous occurrences or deviations from the SAP.

Holly Trejo will also serve as the ECM **Field Manager**, and will oversee field data and sample collection activities.

### 2.1.4 Quality Control Manager, ECM

The project **Quality Control Manager** (QCM), Sandra Maxfield, is responsible for supervising quality control aspects of the fieldwork associated with the project including very specific responsibilities in regard to the acquisition of the project chemical data. The responsibilities of the QCM include:

- Reviewing the SAP/QAPP;
- Assuring chain-of-custody correctness and accuracy;
- Coordinating with project management and the laboratories during sampling events to

ensure compliance with QAPP requirements;

- Recommending corrective action procedures for field activities to maintain QAPP objectives; and
- Ensuring corrective actions are implemented upon identifying out-of-control situations.

### **2.1.5 Field Manager, ECM**

Chris McCormack will conduct the field sampling program with some assistance from Holly Trejo. The Field Manager responsibilities include:

- Conducting field activities in accordance with this SAP/QAPP and associated work plan;
- Using the correct sample collection techniques, sample labeling format, and recording all required information on the field sheets, notebook, and chain of custody documentation;
- Overseeing the transport of all samples to the designated laboratory; and
- Communicating to project personnel/or QCM as needed any conditions in the field that affect sample collection or integrity so that the issues are addressed in a timely manner.

## **3 SAMPLING AND ANALYSIS PLAN**

The SAP presents the technical approach to address the project scope of work and provides a comprehensive description of field activities for sampling and analyzing soil. In addition, the SAP defines the procedures required to ensure that ECM obtains acceptable data of verifiable quality that meet project data quality objectives. The DQOs are discussed in **Section 4.2**.

### **3.1 Purpose and Objectives**

Additional site data are needed to characterize the nature and extent of potential lead contamination at the site. ECM will evaluate the data

- To characterize the extent of lead impacts above background levels,
- To quantify the amount of material that exceeds cleanup goals,
- To assess risks to human health and the environment,
- To establish target risk levels,
- To develop site-specific preliminary remedial goals (PRGs), and
- To evaluate any needed response actions consistent with CERCLA and the National Oil and Hazardous Substances Pollution Contingency Plan (NCP).

### **3.2 Site Description and Background**

DEPO is located along the Middle Fork of the San Joaquin River Valley on the western slope of the Sierra Nevada range between 7,200 and 8,200 feet. It covers approximately 800 acres. The Monument was once part of Yosemite National Park, but discovery of gold in 1905 near Mammoth Lakes prompted a boundary change that left the Postpile on adjacent public land. The monument was set aside by presidential proclamation in 1911.

DEPO's landscape is a result of eruptions and uniform cooling of basalt lava that created an impressive wall of columns which tower 60 feet high. Later, a glacial event exposed the columns and polished smooth the top of this formation, enhancing the pattern of hexagons that resulted from the mineral composition of the lava. Site features are described in greater detail in the Work Plan.

In November 2005, following sandblasting and painting operations at the 100,000 gallon above-ground potable water tank, DEPO collected ten soil samples within the sandblasting containment area to verify the painting contractor's cleanup. The analytical results for the samples indicated a maximum lead concentration of 2,100 milligrams per kilogram (mg/kg) and a minimum concentration of 20 mg/kg. Concentrations of lead in site soils are below the California Human Health Screening Levels (CCHLS) of 3,500 mg/kg for commercial/industrial use. However, the average lead concentrations slightly exceed the Total Threshold Limit Concentration (TTL) of 1,000 mg/kg, as defined in Title 22, California Code of Regulations. The Preliminary Assessment (PA) conducted by Provost and Pritchard Consulting Group (P&P) (P&P, 2008) assigned a Hazard Ranking System score of 9.14; however, no documentation regarding the locations of the 2005 samples was available. **Section 1** of the Work Plan provides a summary of the previous investigation.

A preliminary conceptual site model (CSM) presents the exposure pathways and human receptors for the appropriate contaminated media and transport mechanisms (**Figure 3**).

### 3.3 Data Quality Objectives

Data Quality Objectives (DQOs) define data quality requirements based on the intended use of the data. DQOs are qualitative and quantitative statements that:

- Clarify the sampling and analysis objectives;
- Define the data required for sampling and analysis;
- Determine the appropriate method of data collection; and
- Specify the level of decision errors acceptable for establishing the quantity and quality of data needed to support the project decisions.

The overall QA objective for this project is to develop and implement procedures for obtaining and evaluating data that meet the DQOs. QA procedures ensure field measurements, sampling methods, and analytical data provide information that minimizes sampling error, is comparable and representative of actual field conditions, and that the data generated are technically defensible. Specifically, data must represent an unbiased and precise estimate of the true mean concentration of lead in the upper 0 to 6 inches (Chen, et al, 2000) of soil in areas of the site known or suspected to contain elevated or potentially elevated concentrations.

#### 3.3.1 Data Quality Objectives Process

The EPA's seven-step DQO process (EPA 2006) was used to assist planning for the acquisition of the environmental data at DEPO. The DQO process is used to clarify the study objective, to define the most appropriate data to collect and conditions from which to collect the data, and to specify tolerable limits on decision errors that will be used as the basis for establishing the quantity and quality of data needed to support decision-making. DQOs are used to develop a scientific and resource-effective design for data collection. Each of the seven steps is provided and discussed below in the context of information currently available about DEPO.

##### 3.3.1.1 Step 1: Define the Problem

Characterize impacts and background concentrations of lead in shallow soil near the potable water tank at DEPO to determine acceptable options for remedial actions.

##### 3.3.1.2 Step 2: Establish Decision Statements

The following questions, the consequences of which are described in Step 5 (Develop Decision

Rule) must be answered by the site investigation:

- What is the average background concentration of lead in the shallow soil?
- What is the average concentration of lead in the shallow soil surrounding the potable water tank?

### **3.3.1.3 Step 3: Identify Inputs to the Decision**

Based on the definition of the problem to be addressed (Step 1) and the questions to be resolved (Step 2), the data needs of the study are identified in Step 3 of the DQO process. This step describes the information that must be obtained and the measurements that must be taken to resolve the decision statement (EPA, 2006). The following statements identify inputs necessary to address the decision rules proposed in this project plan:

- Measure the concentration of total lead in the shallow soil surrounding the potable water tank; and
- Measure the concentration of total lead in background (unaffected) shallow soil near the water tank.

### **3.3.1.4 Step 4: Define Study Boundaries**

Spatial and temporal boundaries of the proposed investigation are described in Step 4 of the DQO process. As far as existing data allow, the temporal and lateral boundaries of the problem area are described in the PA. **Figure 2** of the Work Plan depicts site features; however, it is understood that the site boundaries for the investigation will be based on the distribution of paint chips and natural boundaries. Preliminary boundaries are shown in **Figure 3** of the Work Plan. For purposes of this investigation the vertical boundary will be shallow soil within six inches of the surface over the areal distribution of the site.

### **3.3.1.5 Step 5: Establish Decision Rules**

Step 5 of the DQO process defines the parameter of interest, specifies the action level, and integrates study outputs into a single statement that describes the logical basis for choosing among alternative actions. Step 5 essentially delineates the consequences of study results. Decision rules are formulated as "if, then" statements, in which the outcome of the investigation provides direction for the next stage of problem resolution.

Analytical methods proposed in this work plan were selected with detection limits in mind. Chemical and physical data inputs to the decision statements (see discussion under Step 3) will be used to evaluate the following decision rules:

- If true mean lead concentrations in the soil less than 6 inches below the surface at the site exceed background true mean lead concentrations, then concentrations will be evaluated for threat to human health or ecological receptors. If true mean lead concentrations in the surface six inches of soil at the site do not exceed background true mean lead concentrations; then recommendations will be made for no further action;
- If true mean lead concentrations in the soil less than 6 inches below the surface at the site pose a significant threat to human health or ecological receptors, then recommendations will be made for remedial action. If true mean lead concentrations in the surface six inches of soil do not pose a significant threat to human health or ecological receptors, then recommendations will be made for no further action.

### **3.3.1.6 Step 6: Specify the Tolerance Limits on Decision Errors**

Step 6 of the DQO process quantifies the acceptable limits on decision errors. These limits are needed to set the uncertainty that will be acceptable and agreed to by all stakeholders, because there is always some uncertainty in the data (because of errors in sampling and analysis); there is also uncertainty associated with results of statistical tests. If the data are derived from a probability-based sampling design, then statistical analysis can be used to establish the number of samples required. If data are derived from a nonprobability-based (that is, judgmental or authoritative) sampling design, then the use of statistical methods may be limited (EPA, 2001).

For this investigation, we are not interested in small-scale spatial variability at the site. Therefore no weight will be given to areas of suspected high or low concentrations. The sampling design is statistical and the number of samples will be selected to calculate the mean and to quantify the upper confidence level (UCL) of the mean (ITRC, 2012, Section 4).

The quality of analytical data also is assessed as part of this DQO step. Data quality may be specified under measurement quality objectives (MQO). Typically, this quality assessment involves specifying performance criteria in terms of the precision, accuracy, representativeness, completeness, and comparability (PARCC) of the data. These performance criteria provide a measure of how well the established MQOs were met. For this investigation, MQOs for chemical measurements are specified in the QAPP (see **Section 4.5**); the QAPP includes all QA/QC specifications.

### **3.3.1.7 Step 7: Optimize Sampling Design and Implementation**

Limiting sampling error is a critical function of any sampling design and implementation. Sampling error occurs because contaminant concentrations in soil are highly heterogeneous. Typically, only a small number (e.g., 10 - 20) of discrete samples are collected to characterize large areas of suspected contamination. There is a disparity between the magnitude of the mass of the subsample analyzed by the laboratory and the mass of the target area to be investigated or sample volume to be characterized, which can be on the order of 1 in 10 million or more. This increases the chance that the sample misses contamination, which will consequently not be represented in the analytical results at all.

DEPO has selected the incremental sampling methodology<sup>1</sup> (ISM) to characterize the nature and extent of lead contamination at each site. ISM is a suite of planning, sampling, sample preparation, and subsampling techniques that address heterogeneous soil contamination and thereby control sampling errors that may otherwise lead to incorrect decisions. Traditional QA/QC approaches have focused primarily on laboratory procedures, particularly those that take place after a subsample of soil has been extracted, and do not address the major sources of error that occur well before an extract solution is introduced into an analytical instrument. ISM embeds the concept of quality assurance (QA)/quality control (QC) in a meaningful way into planning, design, field sampling and sample processing, as well as laboratory work, by explicitly addressing all of the activities necessary to collect an ISM sample that will be representative of the area(s) of interest. The approach to sample design and implementation is presented below.

## **3.4 Field Methods**

NPS will characterize the nature and extent of lead contamination at the site. ECM field personnel will collect solid matrix samples by hand in the vicinity of the potable water tank. The area surrounding the water tank will be divided into subareas in which potential exposure to lead

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<sup>1</sup> *MULTI INCREMENT*®, a registered trademark of EnviroStat, has also been referenced as incremental sampling methodology (ISM) by analytical laboratories and others.

may be related to historical use and to proximity to the tank. These subareas are referred to as decision units (DUs) and are discussed further below.

Tasks for the sampling and analysis scope of work include:

- Collecting soil samples;
- Chemical testing of samples and complete laboratory QA/QC; and
- Conducting a survey of the number of visible lead particles at the surface.

The following sections describe the implementation of field procedures at DEPO.

### **3.4.1 Incremental Sampling Methodology**

ISM has been selected to characterize each DU at the site because proper execution of the method produces results that are statistically representative of the average concentration within each DU. A DU is an area where a decision is to be made regarding the extent and magnitude of contaminants with respect to the potential environmental hazards posed by the existing or anticipated future exposure to those contaminants. ECM anticipates sampling to include collecting ISM samples from background soils and the following decision units (**Figure 3** of Work Plan):

1. Surface soils within 5 feet of the water tank
2. Surface soils between 5 feet and 15 feet away from the water tank
3. Surface soils between 15 feet and 25 feet away from the water tank

ISM will be implemented in two stages: 1) DU boundary and grid demarcation, and 2) sample collection.

#### DU Boundary and Grid Demarcation

ECM site personnel will delineate the boundaries of each decision unit surrounding the water tank and appropriate information will be transferred to site maps. A circular boundary will be marked at 5 feet, 15 feet and 25 feet from the tank. This will result in three “rings” around the tank representing the three DUs.

Once the DU boundaries are delineated and staked, each DU will be subdivide into 30 approximately equally sized sampling units (SUs) as depicted in **Figure 3** of the Work Plan. Flags or twine will define the edges of each SU and complete the construction of the SUs. Appropriate information of the SU design including orientation, SU dimensions, and nearby features will be recorded in the field notebook. In addition, the completed DU will be photographed for documentation.

#### Sample Collection

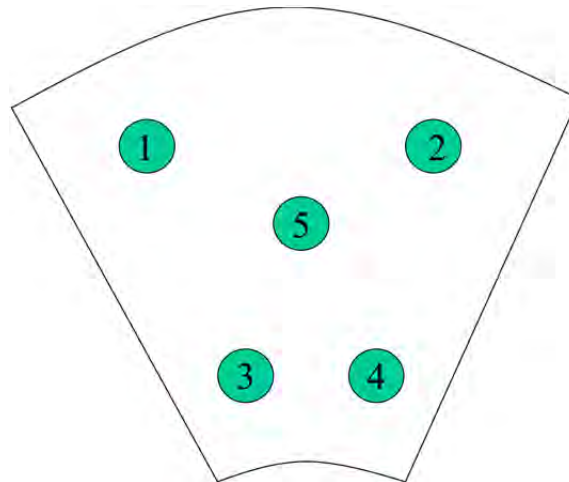
ECM will collect four samples to characterize each decision unit. The systematic random sampling approach will be used to collect samples from each DU. Incremental soil samples will be collected using the following procedure:

1. Establish 5 positions, one at each corner of the SU and one at the center of a SU.



2. Assign the 4 corner positions as 1 through 4 and 5 for the center position as shown below. This configuration will apply to all SUs for all DUs.

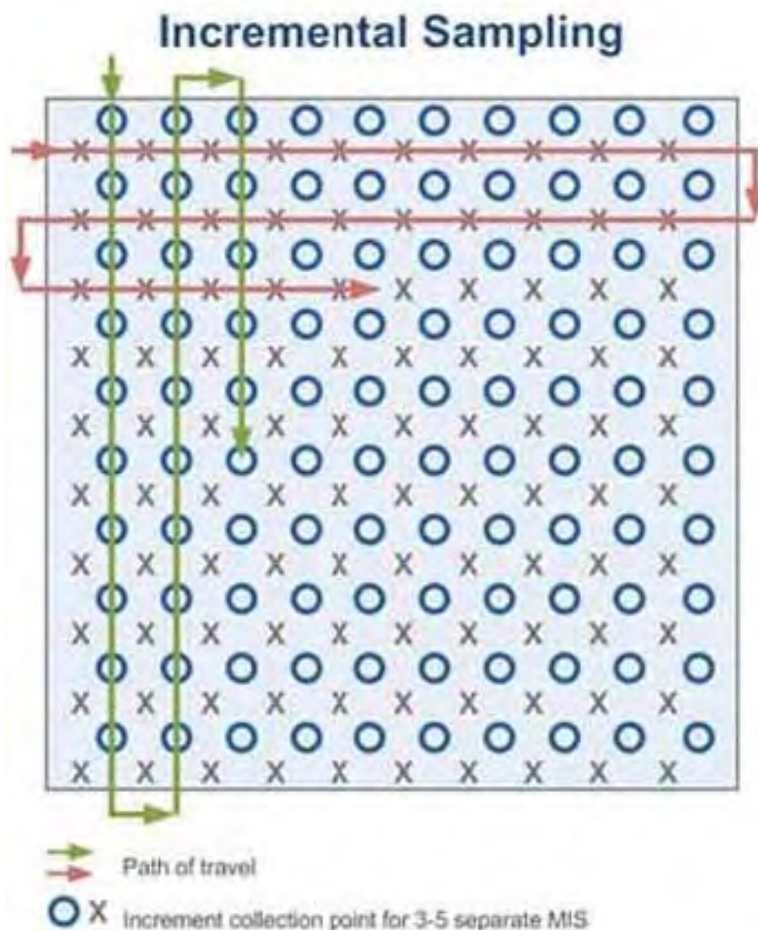
Sampling Unit



3. Roll a single six-sided die to determine the random sample location. For example, if the die turns up "2," soil from position 2 will be collected from all 30 SUs for the first sample. If the die turns up "6", re-roll the die.
4. Roll the die again. For numbers 1 through 3, sampling will proceed clockwise to the next. For numbers 4 through 6, proceed counterclockwise to the next SU. The procedure for the background sample will be discussed in Section 3.4.2.
5. Prepare to collect sample increments by putting on a new pair of disposable nitrile gloves immediately before collecting soil samples.
6. Collect 35 grams of soil from between 0 and approximately 2 inches below ground surface for all DUs with a decontaminated sampling tool. Avoid collecting material larger than 2 millimeters such as stones and roots.
7. Immediately place the soil increment into a clean plastic bag or other appropriate container. A total of at least 1.0 kilograms will be collected from each DU.
8. Proceed to the next SU. It is acceptable to use the same sampling tool between sample increments without decontamination because the sample increments are combined.
9. Repeat steps 6 through 8 to collect the next increment sample until all 30 SUs are completed.
10. Once soil from all 30 SUs is collected, seal the sample container, label the container appropriately and complete the chain-of-custody form.
11. Repeat steps 3 through 10 to collect three additional samples from each DU.
12. Record the sampling activities in the field notebook as follows:
  - Sketch the DU and SU pattern including the 5 positions

- Show the collection locations of all the sample increments on the sketch
- Take photographs of the sampling activities and DU grid, including post-sampling photographs of the area to document the sampling locations.
- Document any deviation from the procedure.

The laboratory will handle all sample sieving, grinding and sub-sampling per the IS method requirements.



### 3.4.2 Background Samples

A clear understanding of the background lead concentrations in soil is important for comparison with site conditions. To obtain defensible background results for the site, ECM will mark the boundary of a background area at each site and grid and sample the background area in the same manner as a DU. The background area will be located upgradient and outside but adjacent to the area potentially affected by the paint chips. Consideration will be given to other features such as roads which may affect the naturally occurring concentration of material in the vicinity of the potable water tank. The areal extent of the background sampling area should be of a scale similar to the DUs.

ISM samples will be collected as described in steps 1 through 12 above. Because the background area may be rectangular in shape, travel will be in the north-south and east-west directions rather than clockwise or counterclockwise. The path of travel for two samples is illustrated below (red represents sample path to the east, green represents path to the south).

### **3.4.3 Quality Assurance/Quality Control Sampling**

Five quality control samples (e.g., field blank, equipment rinsate) will be collected for the site. One equipment rinsate will be collected and submitted from each DU, and one field blank will be submitted for the site.

QA/QC samples for the project will consist of matrix spike/matrix spike duplicate (MS/MSD) samples (prepared by the laboratory), and equipment rinsate of decontaminated multi-use sampling equipment and sample containers. At least one equipment rinsate sample should be collected from the inside of a sample container for the site. A field (water) blank the source of water used to decontaminate equipment will be collected and submitted.

QC samples will be prepared using the same procedures as regular soil samples with regard to sample volume, containers, and preservation. The chain-of-custody procedures for the QC samples will also match those of the field soil samples.

Because volatile analyses will not be performed on any DEPO samples, trip blanks for volatile analysis will not be necessary. Additionally, replicate samples for each DU are included in the sampling procedure; however, at least one replicate from each DU will be submitted to the laboratory as a blind sample.

### **3.4.4 Characterization of Material for Possible Off-site Disposal**

ECM intends to submit all sample material collected to the analytical laboratory, so that no IDW is generated. If IDW is inadvertently generated during site activities, sufficient sample material will be available to analyze for evaluation of disposal of the material off site. If the NPS is considering an excavation and off-site disposal option for the possible removal action, ECM can request that the appropriate analyses including total concentrations of the 8 RCRA metals and TCLP analysis for appropriate metal analytes be performed, if requested by the NPS.

### **3.4.5 Sampling Equipment**

The following equipment will be utilized:

- Sampling tool;
- 5-gallon buckets;
- Decontamination supplies;
- Sample documentation forms and labels;
- Field logbook;
- Wooden stakes, flags, twine;
- Camera;
- GPS unit; and
- Personal protective equipment (PPE).

### **3.4.6 Decontamination Procedures**

Disposable sampling equipment will be used whenever possible to reduce equipment decontamination and expedite sampling. Decontamination will only be required for the sampler.

The following equipment will be necessary to perform sampling equipment decontamination:

- Scrub brushes;
- Hand-held spray bottles;
- Buckets;
- Alconox or other non-phosphate detergent;
- Dilute nitric acid;
- Tap water;
- Plastic bags; and
- Distilled water.

Decontamination procedures for reusable equipment are summarized below:

- Sampling equipment will be decontaminated before initial use, after each sample collected in each DU, and between DUs;
- Sampling equipment will be scrubbed with a stiff brush to remove loose soil and debris;
- Sampling equipment will then be scrubbed with a mixture of Alconox or other non-phosphate detergent and tap water, followed by a tap water rinse;
- Sampling equipment will then be wiped with a solution of 10% nitric acid, followed by a rinse with distilled water using a hand-held spray bottle
- The equipment will then be air dried on plastic sheeting in the field at the sampling location; and
- Following drying, the sampler will be placed in a previously unused plastic bag until it is removed for collection of the next sample.

### **3.4.7 Investigation-Derived Waste Handling**

Investigation-Derived Waste (IDW) generated during field sampling will include disposable sampling equipment, PPE, and decontamination fluids. Decontaminated disposable sampling equipment and PPE will be placed in plastic trash bags and disposed of as municipal waste at an off-site facility. Wash and rinse water from the decontamination of small equipment will be containerized in a 5-gallon bucket or other container as appropriate and relinquished to DEPO for proper disposal. No hazardous wastes will be generated.

### **3.4.8 Particle Survey**

ECM will attempt to survey the number of visible lead particles at the surface, and provide an estimate per square foot. Estimates will be obtained from within each DU and within the background area. The NPS will use this information to assess potential condor lead particle ingestion, as well as to quantify lead particles for remedial soil screening, if warranted. ECM will:

- Collect a grab surface soil (within 2 inches of the surface) from the center of every other SU for DU1.
- Screen soils with a #40 sieve prior to sampling to remove visible lead particles and provide particle count data.

- Examine the material remaining in the screen to determine the number of lead particles.
- Estimate particles per square foot, based on the area of the DU.
- Repeat for the remaining DUs and for the background area.

### **3.4.9 Field Notebooks**

Field personnel will record all information pertinent to the sampling program in a field notebook and/or a field sampling sheet. Each page of field notebook will be initialed and dated by the person making the entries. Notebooks are accountable field documents and serve as a chronological representation of the field activities. Sufficient detail will be included in the notebook to summarize field activities without relying on the recorder's memory. If any corrections are necessary in the notebook, the error will be lined out with a single line and the field personnel will initial the correction. At the end of the field day, blank space in the field notebook will be lined out, initialed, and dated. Examples of typical field notebook entries include the following:

- Date, times when field activities start;
- Personnel present;
- Weather conditions;
- Field measurements, activities, and observations;
- Scope of work for the day;
- Health and safety briefing;
- General sample information (sample ID, time, containers);
- Conditions that may impact sampling;
- Near misses, safety concerns;
- Communication with PM or others, particularly if it changes scope;
- Instrument calibration procedures and frequency;
- Photograph information; and
- Visitors to the site, along with time they arrive and leave.

Information recorded on the field sampling sheet includes:

- Referenced sampling location description (in relation to a stationary landmark) and maps;
- Media sampled;
- Sample collection methods and equipment;
- Date and time of sample collection;
- Types of sample containers used;
- Sample identification and cross-referencing;
- Sample types and preservatives used;
- Analytical parameters;

- Sampling personnel, distribution, and shipping information; and
- Location sketches.

### **3.4.10 Photographs**

Color photographs taken during the sampling activities (at least one photograph at each DU location) will be numbered to correspond to field notebook or photograph log sheet entries. The name of the photographer, date, time, sampling location, and photograph description will be entered sequentially in the field notebook or photograph log as photographs are taken.

## **3.5 Laboratory Analytical Method**

Based on the purpose and objectives of the sampling effort described in Section 2 of the Work Plan and **Section 3.1** of this SAP/QAPP, ECM will submit soil samples for laboratory analysis for lead by EPA Method 6010B.

## **3.6 Sample Containerization and Handling**

This section discusses the requirements for documenting the field work, including sample naming protocol, field notebooks, chain of custody and details on the sample collection, preservation, handling, shipment, and holding times that must be followed during the project. These procedures were developed in accordance with EPA guidance (EPA, 2001) and SW-846 criteria (EPA, 1997).

### **3.6.1 Sample Designation**

A sample numbering scheme has been developed that allows each sample to be uniquely identified and provides a means of tracking the sample from collection through analysis. The numbering scheme indicates the sample type and location. The unique sample number will be entered on sample labels, field sample sheets, COC forms, and other records documenting sampling activities. Self-adhesive sample labels will be affixed to each sample container. The sample identification system will use the following four-part code: X-Y-Z

Where:

X = Project identification (DEPO = Devils Postpile National Monument)

Y = Decision Unit (01 through 03; BG = background, DP = Duplicate)

Z = This is an arbitrary sample number beginning at 101, sequentially assigned to each sample, including QC samples.

For example, the first soil sample, collected from DU 1, will be labeled:

DEPO-01-101

### **3.6.2 Sample Labeling and Containers**

Self-adhesive sample labels will be printed by ECM or provided by the laboratory and affixed to each sample container. The sample label will be completed in indelible ink and will include the following information:

- Project name (DEPO);
- Laboratory identification number;

- Date and time of sample collection;
- Sampler's initials;
- Sample type;
- Preservative used; and
- Analyses requested.

Sample labels will be affixed to the sample containers. New sample containers (plastic bags or other appropriate containers) will be used for each sample.

### **3.6.3 Sample Preservation and Transportation**

Soil samples for lead analysis will be stored at room temperature and do not require special preservation. All samples will be packaged and labeled for shipment in compliance with current regulations. Each sample container will be sealed with packing tape or duct tape and a custody seal will be properly placed across two sides of the lid. If shipped, the COC will be placed inside a sealable plastic bag and taped to the inside of the shipping container. The shipping air bill will be securely attached to the exterior of the bucket. Sample transportation will be initiated to provide adequate time for the project laboratory to meet required holding times. The project laboratory will carry out the chemical analyses and is responsible for storing the samples in a secure location and following all COC procedures.

### **3.6.4 Chain-of-Custody Procedures**

COC procedures establish the documentation necessary to trace sample possession from time of collection through sample analysis and disposition. A sample is in the custody of a person if any of the following criteria are met:

- The sample is in a person's physical possession;
- The sample is in a person's view after being in his or her physical possession;
- The sample was in a person's physical possession and was then locked up or sealed to prevent tampering; and
- The sample is kept in a secured area.

The sample collector will complete a COC to accompany each sample delivery container (cooler) and will be responsible for shipping samples to the laboratory. The sample collector will provide the project name and the sample collector's signature as header information on the COC record.

For each sample, the sample collector will record on the COC the following:

- Project name (DEPO);
- Sample identification ;
- Date and time of sample collection;
- Signature of sampler(s);
- Sample type (solid);
- Number of sample containers;
- Preservative used (N/A if not applicable); and

- Analyses requested.

When shipping the samples, the sample collector will sign the bottom of the COC form and enter the time (24-hour) and date that the samples were relinquished. If applicable, the sample collector will enter the carrier name and air bill number on the form. The original signature copy of the COC record will be enclosed in a plastic bag and secured to the inside of the cooler lid. An example COC is presented in **Appendix A**.

## 4 QUALITY ASSURANCE PROJECT PLAN

The QAPP provides a consistent and detailed framework of policies, procedures, functional activities, and organization to support additional site characterization sampling and analysis at the DEPO potable water tank site. The QAPP outlines the QA program and QC procedures that will help to verify and maintain a level of performance required to meet the project objectives. In addition, the QAPP provides specific descriptions of how the laboratory will implement the QA program.

The QAPP follows and substantially conforms with the project scope and with the following documents to the extent applicable:

- U. S. Environmental Protection Agency, Guidance for the Data Quality Objectives Process, EPA QA/G-4, August 2000;
- U. S. Environmental Protection Agency, EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, March 2001; and
- U.S. Environmental Protection Agency, Guidance for Quality Assurance Project Plans, EPA QA/G-5, December 2002.

### 4.1 QAPP Objectives

This QAPP was prepared to assure that the data collected during field sampling and laboratory analysis is precise, accurate, representative, complete, and comparable to actual site conditions. In addition, the enforcement of this QAPP will confirm that the data collected meets the criteria of technical project procedures during sample collection, sample analysis, and data evaluation.

The objectives of the QAPP are as follows:

- Provide a consistent framework for generating analytical data;
- Address the quality of analytical systems used to perform data collection;
- Discuss procedures that demonstrate that the analytical systems are in control;
- Discuss procedures that limit the effect of non-laboratory activities on analytical data;
- Discuss record-keeping procedures commensurate with project data uses; and
- Provide for generation and documentation of data of known and acceptable quality.

#### 4.1.1 Analytical Laboratories

TestAmerica Sacramento, a California-Certified Environmental Testing Laboratory pursuant to the provisions of the California Environmental Laboratory Improvement Act (Health and Safety Code [HSC]) (Appendix B) performs analyses for EPA method 6010B. TestAmerica Sacramento will also perform the ISM sample preparation. The address and phone number for the TestAmerica Sacramento is:



TestAmerica Sacramento  
880 Riverside Parkway  
West Sacramento, CA 95605  
Telephone: 916.373.5600  
Facsimile: 916.372.1059

TestAmerica's Laboratory Quality Assurance Manual (LQM) is presented in **Appendix B**. The following sections describe the responsibilities of the key laboratory personnel.

#### **4.1.1.1 Laboratory Director**

The Laboratory Director has the ultimate responsibility for the generation of reliable laboratory data. He/she is accountable to his/her General Manager and oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include:

- allocation of personnel and resources;
- ensuring all tasks performed in the laboratory are conducted according to the requirements of the LQM, the Project Technical Profile and/or the appropriate QAPP;
- setting goals and objectives for both the business and employees;
- establishing policies that ensure the quality of analytical services; and
- achieving the financial, business and quality objectives of the laboratory.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced and that the level of service meets ECM's expectations. As such, the Laboratory Director supports a QA Section which has responsibilities independent from sampling and analysis.

The TestAmerica Sacramento **Laboratory Director** is Zan Vicino.

#### **4.1.1.2 Quality Assurance Manager**

The Quality Assurance Manager (QAM) is responsible for ensuring that the laboratory's quality system and LQM meet the requirements set forth in the quality management program, providing quality systems training to all new personnel, maintaining a LQM, and performing or overseeing systems, data, special, and external audits. The QA Manager performs, or supervises, the maintenance of QA records, the maintenance of certifications and accreditations, the submission of monthly QA Reports, and assists in reviewing new work as needed.

The QAM has the full-time responsibility to evaluate the adherence to policies and to assure that systems are in place to produce the level of quality defined in this LQM. Specific responsibilities include:

- Ensuring method validation studies are completed and documented;
- Periodically performing data package inspections;
- Performing data authenticity audits on 100% of analysts and instruments;
- Assisting in the preparation, compilation, and submittal of quality assurance project plans;
- Reviewing program plans for consistency with organizational and contractual requirements and advises appropriate personnel of deficiencies;

- Maintaining QA records;
- Maintaining certifications and accreditations;
- Initiation and oversight of both internal and external audits; documenting root cause investigations for all noted deficiencies; and ensuring timely audit closure;
- Maintaining a corrective action process for internally identified issues and ensures timely closure;
- Monitoring to ensure the documentation of training and method demonstration are current; and
- Facilitating SOP development and document control.

The QA Manager shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QAM is available to any employee at the facility to resolve data quality or ethical issues. The QAM shall be independent of laboratory operations and has an indirect reporting relationship to the director of QA for the laboratory.

The TestAmerica Sacramento **Quality Assurance Manager** is Karla S. Buechler.

#### **4.1.1.3 Laboratory Project Manager**

The Laboratory Project Manager (LPM) responsibilities include:

- preparing the project technical profile which summarizes QA/QC requirements for the project,
- maintaining the laboratory schedule,
- communicating technical requirements to the laboratory, and
- advising the Laboratory, QAM, and Technical Managers of all variances.

The LPM will provide technical guidance and the necessary laboratory-related information to the preparer of project-specific QAPPs and provide peer review of the final document to ensure accuracy of the laboratory information. The LPM will coordinate with the ECM field manager and QCM to ensure the sample containers are prepared and ready for pickup prior to sampling and the samples are received and logged in properly.

The TestAmerica Sacramento **Laboratory Project Manager** is Linda C. Laver.

## **4.2 Laboratory Data Quality**

The laboratory data quality objective is to provide data of known quality to meet the project DQOs. Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards. Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term “analytical quality control”. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements.

### 4.2.1 Quality Assurance Parameters

The QA objectives are defined in terms of precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters. Data that meet the QA objectives and goals will be deemed acceptable. Data that do not meet objectives and goals will be reviewed on a case-by-case basis to ascertain their usefulness. **Section 4.5.1** describes PARCC evaluation. Corrective actions will be implemented to bring data within the QA acceptability goals whenever possible.

The DQOs for this project require the quantitative and qualitative verification of 100% of sample results. Substantiating the results of the data provides confidence in the project data to support decisions regarding physical and/or chemical properties of the samples, verification of contaminant identification, and other decisions relevant to the project.

The parameters of precision, accuracy, and completeness provide a quantitative measure of the statistical significance of the data collected in this field program. The parameters of representativeness and comparability utilize documentation of the field and laboratory procedures to qualitatively evaluate the data. Following the collection and analyses of the samples, a determination will be made whether the DQOs established for this QAPP were satisfied.

## 4.3 Laboratory Analytical Procedures

The laboratory will perform the analytical work required for project sampling activities. The laboratory QA procedures provide rules and guidelines to ensure the reliability and validity of analytical results. They describe the QA and QC procedures the laboratory utilizes during laboratory analyses and reporting to provide data with the quality necessary for the data's intended uses. The QA/QC objectives and procedures, calibration procedures, and performance audit are included in the LQM.

The objectives of the laboratory QA/QC program are to:

- Ensure that procedures are documented, including any changes in administrative and/or technical procedures;
- Ensure that analytical procedures are conducted according to sound scientific principles and are validated;
- Monitor the performance of the laboratory by a systematic inspection program and provide for corrective action, as necessary;
- Collaborate with other laboratories, if needed, in establishing quality levels, as appropriate; and
- Ensure that data is properly recorded and archived.

Laboratory procedures are documented in writing as either Standard Operating Procedures (SOPs) or Standard Analytical Procedures. The laboratory will conduct internal QC procedures for analytical services in accordance with laboratory procedures.

### 4.3.1 Laboratory Analytical and Measurement Procedures

The laboratory analytical program in support of additional site characterization sampling and analysis will include the following parameters:

- EPA 6010B – Lead (Soil)

### **4.3.2 Method Detection Limits, Practical Quantitation Limits, and Instrument Calibration and Maintenance**

The reporting limits used by a laboratory are derived specifically for each laboratory, instrument, and sample matrix. Therefore, instrument calibration is necessary to ensure reliable and accurate test results. Because each laboratory follows a rigorous instrument calibration process, in practice, most reporting limits are associated with the analytical method. Every analytical method results in limits that are typically attained under routine laboratory operating conditions. A discussion of detection and quantitation limits follows.

#### **4.3.2.1 Method Detection Limits**

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

#### **4.3.2.2 Practical Quantitation (Reporting Limits) Limits**

The Practical Quantitation Limit (PQL) (reporting limits [RLs]) are the lowest level that can be reasonably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The PQLs for lead are 5 mg/kg for soil. The MDL is lower than the PQL.

#### **4.3.2.3 Instrument Calibration**

Before the laboratory analyzes samples, they will analyze chemical calibration standards of each target analyte to establish that the instrument is functioning properly with the desired sensitivity as described in the LQM. The calibration standards are injected into the instrument under the same conditions as the samples. The concentrations of the chemical calibration standards are chosen to bracket the optimum range of the method. The operator tunes and optimizes the instruments daily. The operator also maintains a log documenting the tuning and optimization. A maintenance book is kept for each instrument showing description, manufacturer, model number, serial number, date of last problem, maintenance, repair, and minor change of service.

## **4.4 Quality Control Checks**

A series of QC samples, consisting of equipment blanks and duplicates, will be collected in the field and submitted to the laboratory with the field samples for analysis (**Section 3.3.3** of the SAP). The laboratory and the Project Chemist will use these samples to assess the impact of the field-sampling program on data quality and the overall laboratory analytical data quality. Batch laboratory QC samples including laboratory control samples (LCS), laboratory control sample duplicates (LCSD), matrix spikes (MS), laboratory duplicates, and matrix spike duplicates (MSD) will be analyzed in accordance with SW-846 QC protocols and the laboratory SOPs. The batch laboratory QC sample types are described in **Section 4.4.2** below.

### **4.4.1 QA/QC Sampling Frequency**

The following QC samples will be collected during sampling:

- Blind duplicate – one sample from each DU will be submitted as a blind duplicate (*i.e.*, sample identification will not include the DU number)

#### **4.4.2 Internal Quality Control**

Laboratory internal QC checks include:

- Internal system checks, and
- Controlled samples introduced by the laboratory into the sample analysis stream to monitor day-to-day variations in routine laboratory analyses.

The laboratory will use these procedures to validate the data and calculate the accuracy and precision of the chemical analysis program. The overall level of laboratory QC will conform to the minimum required QC protocols of the standard EPA methods, as amended by the laboratory-specific procedures for these methods.

The laboratory's method-specific SOPs will define the types of QC checks required (*i.e.*, laboratory control samples, method blanks, matrix spike/matrix spike duplicates, post digestion spikes, calibration standards, internal standards, surrogate standards, specific calibration check standards, sample dilutions, and/or laboratory duplicate analysis). Laboratory method-specific SOPs also will define the frequency of each QC analysis, the analytes and reference concentrations to be used as controls, and the QC acceptance criteria. The general types of laboratory internal QC checks are summarized below:

##### **4.4.2.1 Laboratory Control Sample**

The laboratory control sample is an interference-free matrix spiked with known quantities of the analytes of interest or specific compounds. The LCS helps identify the accuracy of the analytical method by establishing a basis for the percent recovery of the spiked compounds.

##### **4.4.2.2 Matrix Spike/Matrix Spike Duplicates**

The laboratory creates MS/MSD samples by adding known concentrations of target analytes or specific compounds to an aliquot of the applicable site matrix immediately before extraction and analysis. MS/MSD samples provide information on matrix interferences encountered during extraction, digestion, and analysis (*i.e.*, suppression or enhancement of instrument signal levels). MS samples principally provide a means to evaluate accuracy. A comparison between MS samples and associated MSD samples with a relative percent difference (RPD) of  $\pm 20\%$  indicates analytical precision.

##### **4.4.2.3 Method Blanks**

Method blanks consist of contaminant-free, reagent-water samples spiked with all reagents, surrogates, and internal standards that undergo the entire analytical procedure. Method blanks help reveal system bias introduced in the laboratory. A method blank should have a value below the reporting limit of the constituents of concern but above the minimum detection limit.

##### **4.4.2.4 Surrogate Spikes**

The laboratory prepares surrogate spikes by adding a known amount of analytes, chemically similar to the target analytes, to every blank, sample extract, matrix spike, matrix spike duplicate, and standard. Surrogate spike results help evaluate analytical efficiency and matrix interferences.

#### **4.5 Calculations of Data Quality Indicators**

This section summarizes QA/QC procedures for assessing the validity of the chemical data after sampling and analysis and the format for presenting the results of the QA/QC evaluations in

ECM reports. The Project Chemist will assess QA/QC after the laboratory has compared QC results to the method- and project-specific goals presented in this QAPP.

The QCM (or designated representative) will use the data verification procedures for statistically assessing duplicate and external spike samples that are submitted blind to the analytical laboratories from the field and generated internally by the laboratories in accordance with this QAPP. Blind submittals ensure that the laboratory treats these QC samples the same as other samples and does not subject them to special treatment. In addition, the QCM will evaluate all sample results to determine if reporting detection limit goals (e.g., analytical sensitivity) were met and if test sample dilutions were justified. The purpose of implementing these procedures is to verify that the chemical data generated during the investigation are of acceptable accuracy, precision, and completeness and are representative of site conditions.

#### 4.5.1 Assessment of PARCC

The goals of the assessment of PARCC are the following:

- Determine site-specific PARCC,
- Use PARCC results to identify the limits of data usability, and
- Evaluate these limitations in achieving the program DQOs.

The parameters of precision, accuracy, and completeness provide a quantitative measure of the statistical significance of the data collected in this field program. The parameters of representativeness and comparability utilize documentation of the field and laboratory procedures to qualitatively evaluate the data.

Precision measures the agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision describes the effects that random errors have on analytical measurements. Precision is the degree to which the measurement is reproducible and is usually expressed in terms of relative percent difference or standard deviation.

Accuracy is the degree of agreement of a measurement with an accepted reference or true value. The matrix spike procedure (**Section 4.4.2.2**, above) helps the laboratory determine the accuracy of an analytical procedure. Accuracy represents the impact of systematic errors, or biases, on analytical measurements required to make programmatic decisions.

Representativeness expresses the degree to which sample data accurately and precisely represent actual site conditions. Representativeness involves the selection of analytical methods and sampling protocols and locations such that results truly represent the media being sampled (*i.e.*, soil) and the conditions being measured. It is the qualitative parameter concerning the proper design of the sampling program.

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected and needed to meet the project data goals. The completeness goal for this project is 90 percent.

- Field completeness is assessed by comparing the number of samples collected to the number of samples planned.
- Analytical completeness includes acceptable data completeness and quality data completeness. Acceptable data is defined as either data that passed all QC criteria or data that received qualification due to QC and/or method limitations, but were not qualified as rejected (R). Quality data is defined as data that passed all QC criteria

(receiving neither an estimated (J) nor “R” qualification). Acceptable completeness is assessed by comparing the total number of samples with valid analytical results (as described) to the number of samples collected.

Comparability is a qualitative measure of the confidence with which one data set will match another data set except with regard to time and space. Sampling programs achieve comparability by incorporating standard methods to collect and analyze representative samples and reporting the resulting data in standard units.

The QCM and/or Project Chemist will evaluate chemical data derived from the investigation based on PARCC assessment for both the laboratory analytical and field sample collection programs. To address these issues, a combination of qualitative evaluations and comparisons to project QA objectives will be used to check the quality of the chemical data. A quantitative evaluation will be made of precision, accuracy, and completeness. A qualitative evaluation will be made of representativeness and comparability based on assessment of precision, accuracy, and completeness. The results of the evaluations will not be used to eliminate data from the database. Comparisons of internal laboratory QC samples to project QA goals (e.g., precision and accuracy goals specified by the methods and this QAPP) will support the validation of each laboratory’s analytical procedures.

The Project Chemist and/or QCM will use PARCC parameters to determine if data meet the project’s DQOs. The Project Chemist will assign database qualifiers to chemical data that do not meet relevant QC criteria. Data qualifiers indicate the degree to which the sample data conform to QC requirements during the data review process. The following sections present procedures for evaluating the PARCC of data derived from the investigation.

#### **4.5.1.1 Precision**

The laboratory, Project Chemist, and/or QCM will calculate precision for sample data by evaluating data from field duplicate samples, LCS/LCSD, and MS/MSD duplicate samples, as follows:

- Tabulate duplicate data and calculate the absolute value difference, average, and Relative Percent Difference (RPD) as shown below for each duplicate pair:

$$RPD = \frac{(x_1 - x_2) \times 100\%}{\bar{x}}$$

Where:

$x_1$  = concentration for Sample 1 of duplicate,  
 $x_2$  = concentration for Sample 2 of duplicate, and  
 $\bar{x}$  = mean of Samples 1 and 2;

- RPDs will not be calculated in cases where:  
One or both analytes of the duplicate pair are reported as not detected (ND),  
when a duplicate pair are within a factor of three of each other and the lowest value is  $\leq$  five times (5x) the reporting limit (EPA, 2004);
- Identify duplicates that exceed the project (method) precision goal of  $\pm 20\%$ ; and
- Qualitatively evaluate the significance of data that fall outside project precision goals. Usability of data outside of project goals for precision depends on the degree of QC exceedance, the presence of potential high or low sample result bias, the significance of associated sample results compared to action levels, and if the sample is critical to the investigative findings. If data quality problems arise, the Project Chemist and/or QCM

will notify the analytical laboratory for corrective action, as appropriate. Data will not be removed from the database solely as a result of these procedures, and QC samples will not be used to alter or correct analytical data. Data will be flagged with appropriate notation.

#### **4.5.1.2 Accuracy**

The laboratory, Project Chemist, and/or QCM will calculate the accuracy for sample data by evaluating data from blanks and MS QC samples, as described below.

##### **Blanks**

- Tabulate the data from the blank samples.
- Identify any blank samples in which chemicals are detected.
- If no chemicals are detected in any blank samples, enter the tables into a summary report.
- If any chemicals are found in field and laboratory blank samples, assess the compound(s), concentration(s), and field data for that period of time for potential problems with data interpretation. Data will not be removed from the database on the basis of chemicals detected in field or laboratory blank samples, nor will data from QC samples be used to alter or correct sample analytical results. Appropriate notations will be made in the following database reports.
- Inorganic laboratory method blanks: If metals are detected in method blanks at concentrations less than five (5) times the reporting limit, those metals detected in associated environmental samples will qualify as not detected.
- Field blanks (field, equipment or rinseate, and trip blanks): if common laboratory contaminants appear in field blanks, associated environmental samples will only count as positive if the concentration exceeds ten (10) times the maximum concentration in the blank(s). Other compounds will be treated similarly, except that the allowed level will be five (5) instead of ten (10) times the concentration detected in the field blank(s).

##### **Spikes**

The laboratory will implement procedures for assessing MS/MSD and surrogate spike samples are as follows:

- Tabulate spike sample data and calculate the percent recovery as shown below for each sample:

$$\text{Percent Recovery} = \frac{(T - X) \times 100\%}{A}$$

Where:

T = total concentration found in spiked sample,

X = original concentration in sample matrix prior to spiking, and

A = actual spike concentration added to sample;

- Identify spikes that exceed the project (method) percent recovery goals for accuracy; and
- Qualitatively evaluate the significance of data that fall outside the project goals for accuracy. Usability of data outside of project goals for accuracy depends on the degree



of QC exceedance, presence of a potential high or low sample result bias, significance of associated sample results compared to action levels, and importance of the sample to the investigative findings. If MS/MSD do not meet project requirements, the Project Chemist or QCM will notify the laboratory, evaluate the data from that period of time for the compound that exceeds the limits, and take corrective action as appropriate. Data will remain in the database as a result of these procedures with no alterations or corrections using QC samples. Instead, the appropriate notations will note qualified data.

#### **4.5.1.3 Representativeness**

The Project Chemist and/or QCM will qualitatively assess representativeness of data by evaluating whether or not sample collection and analytical procedures described in this SAP and QAPP were followed. Specifically, they will review the site sampling layout, including sampling locations, frequencies, and timing, as well as precision and accuracy information developed from the evaluation of QC samples.

#### **4.5.1.4 Completeness**

The completeness goal for this project is 90 percent. The Project Chemist and/or QCM will calculate overall completeness of the sample data using the following equation:

$$C = \frac{V}{T} \times 100\%$$

Where:

C = percent completeness of analytical effort,

V = amount of valid data obtained, and

T = amount of samples collected and analyzed.

Completeness calculations will use validated data. Data that are rejected by external validation processes will count against completeness criteria. QC parameters evaluated to assess completeness include holding times, surrogates, laboratory and field duplicates, RPD, MS/MSD for percent recovery and RPD, and LCS for percent recovery. Samples results that do not meet relevant QC criteria due to substantiated matrix effects, and/or are re-analyzed past holding time due to QC corrective action, and/or are "J" qualified because the sample results are below the reporting detection limit, will be considered usable and will not count against the completeness assessment.

#### **4.5.1.5 Comparability**

The comparability evaluation will include a qualitative assessment of analytical techniques, data quality, and sampling design. The Project Chemist and/or QCM will assess comparability of the analytical techniques by referencing the analytical data reports submitted by the laboratory. Specific items to be evaluated include sampling and analytical method equivalency, preservation methods, detection limits, reporting units, equivalent laboratory facilities and personnel, QA/QC programs, DQOs, and precision and accuracy estimates. If the above factors are generally equivalent, the data sets will be considered comparable.

### **4.6 Corrective Action**

The QCM will detect problems or potential system problems by performing calibration check samples, QC samples, daily performance audits, and QA audits. The QCM will immediately discuss these problems and possible solutions with the Laboratory Director and lab supervisors.

### 4.6.1 Laboratory Situations

The QAM will initiate the need for corrective action resulting from evaluation of QA/QC results in consultation with the project QCM. Corrective action may include, but is not limited to:

- Re-analyzing the samples, if holding-time criteria permit;
- Evaluating and amending sampling and analytical procedures;
- Accepting data with an acknowledged level of uncertainty; and
- Resampling and analysis, if the completeness of the data set or intended use of the data are recognized during a preliminary review to be insufficient to meet program DQOs.

If the Project Chemist and/or QCM deem the above corrective actions unacceptable, they will select an alternate laboratory to perform necessary or appropriate verification analyses in consultation with the PM.

### 4.6.2 Immediate Corrective Action

Any equipment and instrument malfunctions will require immediate corrective actions. The laboratory QC charts are working tools that identify appropriate immediate corrective actions to be taken when instruments and equipment exceed a control limit. The operator should note any corrective actions in field or laboratory logbooks, but no other formal documentation is required unless further corrective action is necessary.

### 4.6.3 Long-Term Corrective Action

Standard QC procedures, control charts, and/or performance or system audits may identify the need for long-term corrective action. Any quality problem that immediate corrective action cannot solve will fall into the long-term category. The essential steps in a long-term corrective action system are:

- Identification and definition of the problem;
- Investigation and determination of the cause of the problem;
- Determination and implementation of a corrective action to eliminate the problem; and
- Verification that the corrective action has eliminated the problem.

Documentation of the problem is important in corrective action. The responsible person may be an analyst, the QAM, the QCM, or the PM. In general, the QCM will investigate the situation and determine who will be responsible for implementing the corrective action. For field activities, the QCM will document the required corrective action.

### 4.6.4 Out-of-Control Situations

An out-of-control situation arises when a value falls outside the control limits or when statistical testing (per laboratory quality assurance documents in **Appendix B**) classifies a value as an outlier. Failure to meet calibration criteria, record keeping omissions, improper sampling technique, and improper storage or preservation of samples are all conditions that affect data quality and require investigation and correction. If an out-of-control situation arises, the Project Chemist will notify the QCM and ask the laboratory to take immediate action to find the problem, recalibrate, and re-analyze the samples.

#### **4.6.5 Laboratory Corrective Procedures**

When the laboratory quality assurance representative identifies an out-of-control situation, the analyst, laboratory supervisor(s), and laboratory manager will investigate to determine the cause, notify and consult with the QCM and document the actions taken. The Project Chemist will discard any data acquired concurrently with this condition and direct the laboratory to re-analyze samples unless the investigation of the problem proves that the analysis was in control.

After the laboratory institutes corrective actions, the laboratory will rigorously check the system's performance before continuing sample analysis. The laboratory will not resume analysis if the calibration check samples are outside of the EPA limits. They must diagnose the problem, fix the system, and re-check the calibration before resuming analysis. Finally, the laboratory will document these corrective actions associated with the project and maintain the records in their maintenance book.

### **4.7 Data Evaluation, Recording, Reduction, and Reporting**

The Project Chemist and/or QCM will manage, distribute, and preserve data collected during implementation of the groundwater and solid mine waste sampling event at DEPO to substantiate and document that data are of known quality and are properly maintained.

#### **4.7.1 Data Evaluation**

Data review is an essential tool to assure the validity of the reported data; therefore, all analytical data must be completely and thoroughly evaluated through three levels of documented review. Data quality is the primary responsibility of the laboratory chemist performing the analysis, who will ensure quality through adherence to laboratory and methodological QC and performance parameters. The QCM is responsible for ensuring that the data complies with the procedural guidelines of the method and any project specific requirements. Each level of review is documented in the appropriate checklist according to the applicable laboratory SOP.

##### **4.7.1.1 Internal Laboratory Analytical Data Review**

The laboratory will review analytical data to assure that results for investigative and QC samples meet EPA functional guidelines or method-specified criteria. Laboratory review of analytical data will conform to EPA guidance document SW-846 and laboratory SOPs. For target analytes without specific QC criteria per EPA guidance documents, the laboratory will base the QC criteria on their own method-specific requirements.

On the basis of a comparison of the data with established QC limits, the laboratory will assign qualifiers that are consistent with laboratory method-specific SOPs and/or EPA functional guidelines to the data as necessary to further evaluate a reported result. The qualifiers will be attached to the data whenever they appear in hard copy or computerized form to assure that data users are aware of the quality and limitations of the data.

##### **4.7.1.2 External Laboratory Data Review**

The Project Chemist will review laboratory analytical data to independently verify the laboratory results. The laboratory data package will be subjected to 100% data review upon delivery from the contract lab no later than 30 calendar days after receipt of the last analytical result from the laboratory. The data review process will address the following elements of the laboratory data package:

- case narrative;
- data completeness;
- holding times;
- chain-of-custody;
- method blanks;
- laboratory control samples;
- matrix spike/matrix spike duplicate;
- surrogate recoveries; and
- field duplicate results.

#### **4.7.2 Data Reporting**

The laboratory data report will include a cover letter with a summary of the analytical data presented. The report will include information concerning the media sampled, the sample quantities, the purpose of the sampling, the dates of the sampling event, and the identity of the sampler. The laboratory will provide a data package of sufficient quality and completeness to allow data evaluation by the Project Chemist. The deliverables will include, but not be limited to the following, where appropriate:

- Letter summarizing the analytical procedures required for the samples received;
- Case narrative summarizing the appropriate information detailing any problems with the analysis or the laboratory quality control and the corrective action taken;
- Package checklist;
- Sample results summary package;
- Batch specific QC results including LCS, MS/MSD, method blank data, surrogate recoveries, and inorganic sample duplicates;
- Definitions of lab qualifiers; and
- Original Chain-of-Custody Record.

#### **4.8 Data Acquisition Requirements**

Data acquired through sample analyses will be reported (1) by following formats established by the method and (2) within the required deliverable schedule. All data from project laboratories will be presented with QA summary forms that allows for independent review of data quality. Full CLP-like data packages may be requested at a later date.

All field information will be recorded on appropriate field forms and will be reviewed for accuracy and completeness.

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EPA. 1997. *Test Methods for Evaluating Solid Waste/Chemical Methods, Laboratory, Volume JA through 1C, and Field Manual, Volume 2. SW-846, Third Edition (Revision III)*. Office of Solid Waste and Emergency Response.

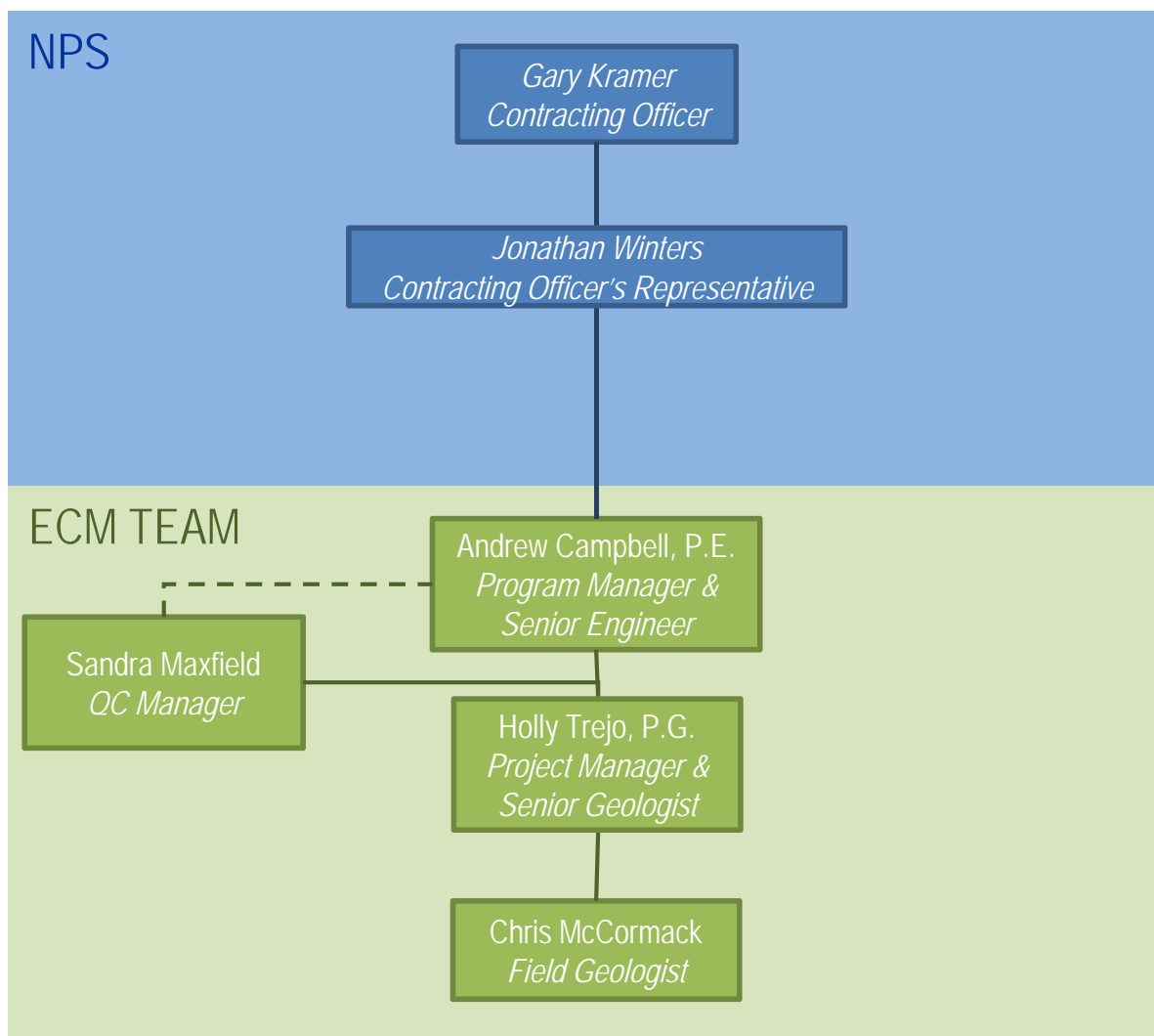
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EPA. 2002. *Guidance for Quality Assurance Project Plans*, EPA QA/G-5, December 2002.

Interstate Technology and Regulatory Council (ITRC), 2012. *Incremental Sampling Methodology*, February 2012.

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## **Figures**



Devils Postpile National Monument  
Madera County  
Mammoth Lakes, CA 93546

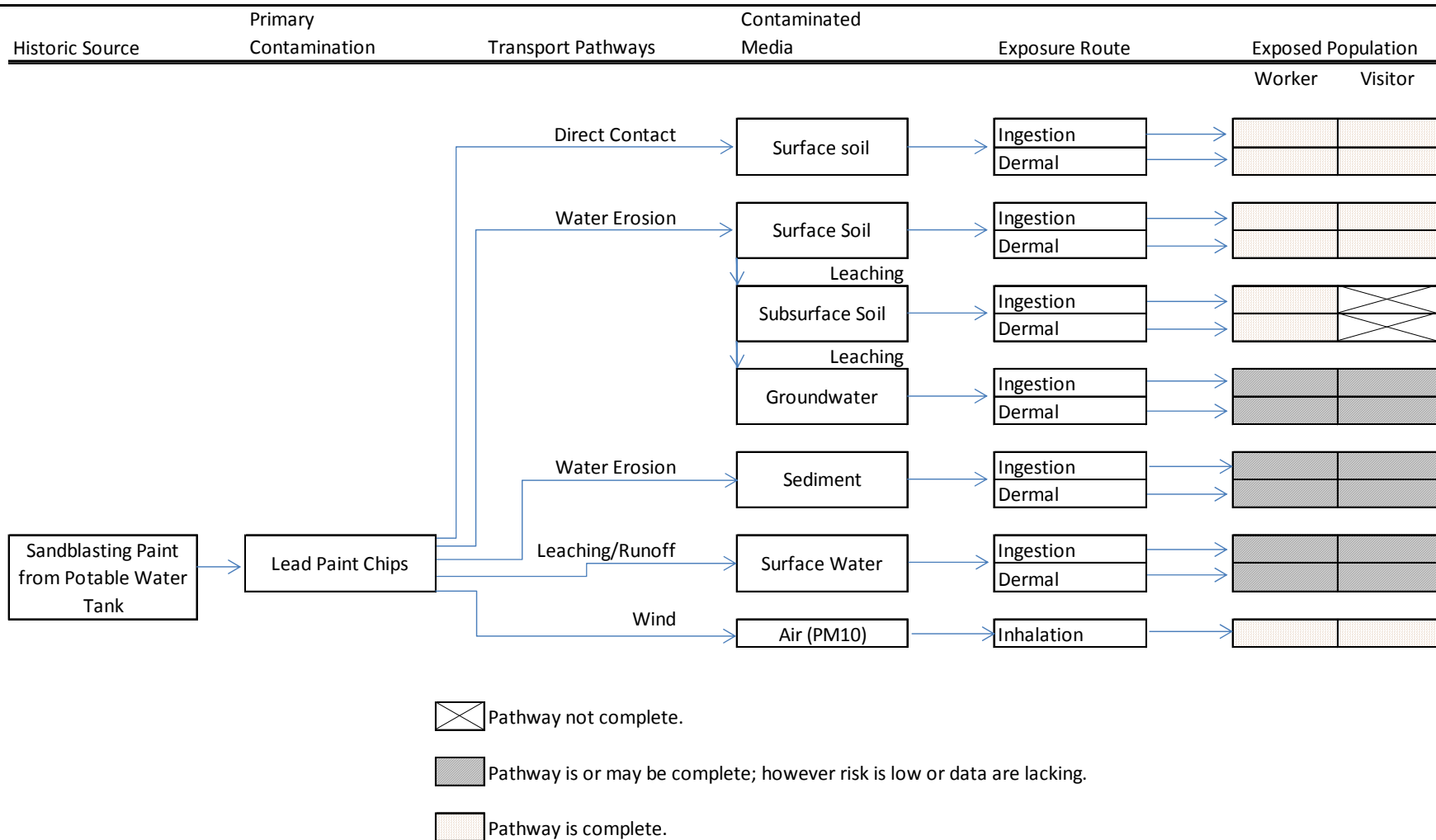


**ENVIRONMENTAL COST MANAGEMENT, INC.**  
*Managing Cost and Liability*  
3525 Hyland Avenue, Suite 200 • Costa Mesa, CA 92626  
Tel: (714) 662-2759 • Fax: (714) 662-2758

Project Organization

Figure

2





## **Appendices**

## **Appendix A: Field Sampling Forms**



ENVIRONMENTAL COST MANAGEMENT, INC.  
*Managing Cost and Liability*

3525 Hyland Avenue, Suite 200  
Costa Mesa, California 92626  
Main: (714) 662-2759 Fax: (714) 662-2758  
[www.ecostmanage.com](http://www.ecostmanage.com)

## PHOTOGRAPHIC LOG

Photo #	Description
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**TestAmerica Sacramento**

880 Riverside Parkway

West Sacramento, CA 95605  
phone 916.374.4378 fax 916.372.1059

**Chain of Custody Record**

**TestAmerica Laboratories, Inc.**
**Regulatory Program:** ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

<b>Client Contact</b>		<b>Project Manager: Holly Trejo</b>		<b>Site Contact: Holly Trejo</b>		<b>Date:</b>		<b>COC No:</b>	
Environmental Cost Management, Inc.		<b>Tel/Fax: 510-964-4399</b>		<b>Lab Contact: Linda C. Laver</b>		<b>Carrier: ECM</b>		_____ of _____ COCs	
3525 Hyland Anve, Suite 200		<b>Analysis Turnaround Time</b> <input type="checkbox"/> CALENDAR DAYS <input type="checkbox"/> WORKING DAYS TAT if different from Below _____ <input checked="" type="checkbox"/> 2 weeks <input type="checkbox"/> 1 week <input type="checkbox"/> 2 days <input type="checkbox"/> 1 day		<div style="display: flex; flex-direction: column; align-items: center;"> <div>Filtered Sample (Y / N)</div> <div>Perform MS / MSD (Y / N)</div> <div>Total Lead EPA 200.7</div> <div>Total Lead EPA 6010 B</div> </div>				<b>Sampler:</b> <b>For Lab Use Only:</b> Walk-in Client: _____ Lab Sampling: _____  Job / SDG No.: _____	
Costa Mesa, CA 92626									
(714) 662-2759 Phone									
(714) 662-2758 FAX									
Project Name: NPS-DEPO									
Site: DEPO Water Tank									
P O #									
<b>Sample Identification</b>		<b>Sample Date</b>	<b>Sample Time</b>	<b>Sample Type</b> (C=Comp, G=Grab)	<b>Matrix</b>	<b># of Cont.</b>			Sample Specific Notes:
					Soil				
<b>Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other</b> _____									
<b>Possible Hazard Identification:</b> Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample. <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown							<b>Sample Disposal ( A fee may be assessed if samples are retained longer than 1 month)</b> <input type="checkbox"/> Return to Client <input type="checkbox"/> Disposal by Lab <input type="checkbox"/> Archive for _____ Months		
<b>Special Instructions/QC Requirements &amp; Comments:</b>									
Custody Seals Intact: <input type="checkbox"/> Yes <input type="checkbox"/> No		Custody Seal No.:		Cooler Temp. (°C): Obs'd: _____		Corr'd: _____		Therm ID No.: _____	
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Relinquished by:		Company:		Date/Time:		Received in Laboratory by:		Company:	

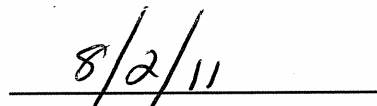
## **Appendix B: Laboratory Quality Assurance Manual (LQM)**


# Quality Assurance Manual

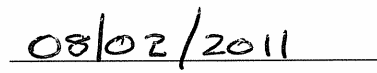
**TestAmerica West Sacramento**  
**880 Riverside Parkway**  
**West Sacramento, CA 95605**  
**Phone No. (916) 373-5600**  
**Fax No. (916) 372-1059**  
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## Approval Signatures:

  
Laboratory Director – Karla Buechler

  
Date

  
Quality Manager - Douglas Weir

  
Date

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## REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

## REFERENCED LABORATORY SOPs

SOP Reference	Title
WS-PQA-013	Procedures to Address Customer Complaints
WS-QA-0050	Management of Change
WS-QA-0009	Document Archiving
WS-QA-0022	Employee Orientation and Training
WS-QA-0021	Preparation and Management of Standard Operating Procedures
WS-QA-0006	Method Detection Limits (MDL) and Instrument Detection Limits (IDL)
WS-PQA-0011	Manual Integration Documentation Procedures
WS-QA-0018	Subsampling and Compositing of Samples
WS-QA-0003	Sample Receipt and Procedures



## SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

### 3.1 Introduction and Compliance References

TestAmerica West Sacramento's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water*, EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water*, Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water* (EPA 815-R-05-004, January 2005)
- Statement of Work for Inorganics & Organics Analysis, SOM, ISM, DLF and CBC, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- APHA, *Standard Methods for the Examination of Water and Wastewater*, 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup>, 21<sup>st</sup>, and on-line Editions.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan* (QAPP), Version 4.0.02, May 2006.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

### **3.2 Terms and Definitions**

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

### **3.3 Scope / Fields of Testing**

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

### **3.4        Management of the Manual**

#### **3.4.1      Review Process**

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. WS-QA-0021).

## **SECTION 4. MANAGEMENT REQUIREMENTS**

### **4.1 Overview**

TestAmerica West Sacramento is a local operating unit of TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica West Sacramento is presented in Figure 4-1.

### **Roles and Responsibilities**

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

#### **4.1.1 Additional Requirements for Laboratories**

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's West Sacramento laboratory.

#### **4.1.2 Laboratory Director / Technical Director**

TestAmerica West Sacramento's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

#### **4.1.3 Quality Assurance (QA) Manager or Designee**

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.

- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025.

#### **4.1.4 Technical Manager (Operations Manager) or Designee**

The Operations Manager has the responsibility for the day to day operations of the analytical staff within the laboratory. **The Operations Manager is responsible for compliance with the ISO 17025 Standard.** The Operations Manager reports directly to the Laboratory Director. The Operations Manager schedules analytical operations, ensures that the laboratory meets quality requirements, investigates technical issues as they arise, and performs other tasks as required by the NELAC standards.

#### **4.1.5 Manager of Customer Services**

The Manager of Customer Services has the responsibility for the day to day operations of the client services staff, which includes the Project Management and other administrative groups within the laboratory. The Manager of Customer Services reports directly to the Laboratory Director. The Manager of Customer Services has signature authority for contracts for laboratory services (as detailed in TestAmerica policy), and for laboratory reports.

#### **4.1.6 Project Manager**

Project Managers are a liaison between the laboratory's clients and the analytical staff. They report directly to the Manager of Customer Service. The Project Managers have signature authority for final reports, and review project data packages for completeness and compliance with client needs and quality requirements.

#### **4.1.7 Project Administrator**

Project Administrators are a liaison between the laboratory's clients and the analytical staff. They report directly to the Manager of Customer Service. The Project Administrators review project data packages for completeness and compliance with client needs and quality requirements.

#### **4.1.8 Department Manager, Team Leader, or Supervisor**

Department Managers report directly to the Operations Manager. They supervise the daily activities of analysis with a given laboratory area, and either oversee the review and approval, or perform the review and approval of all analytical data within that area.

#### **4.1.9 Analyst**

Analysts report to their respective Department Managers. They perform sample analyses and generate analytical data in accordance with documented procedures.

#### **4.1.10 Sample Custodian**

The Sample Custodian ensures the implementation of proper sample receipt procedures, including maintaining chain-of-custody. The Sample Custodian logs samples into the LIMS and ensures that all samples are stored appropriately.

#### **4.1.11 Report Production Staff**

The Report Production Staff accurately generates and compiles analytical reports and the associated deliverables as required by the client.

#### 4.1.12 Quality Assurance Staff

The QA Staff has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

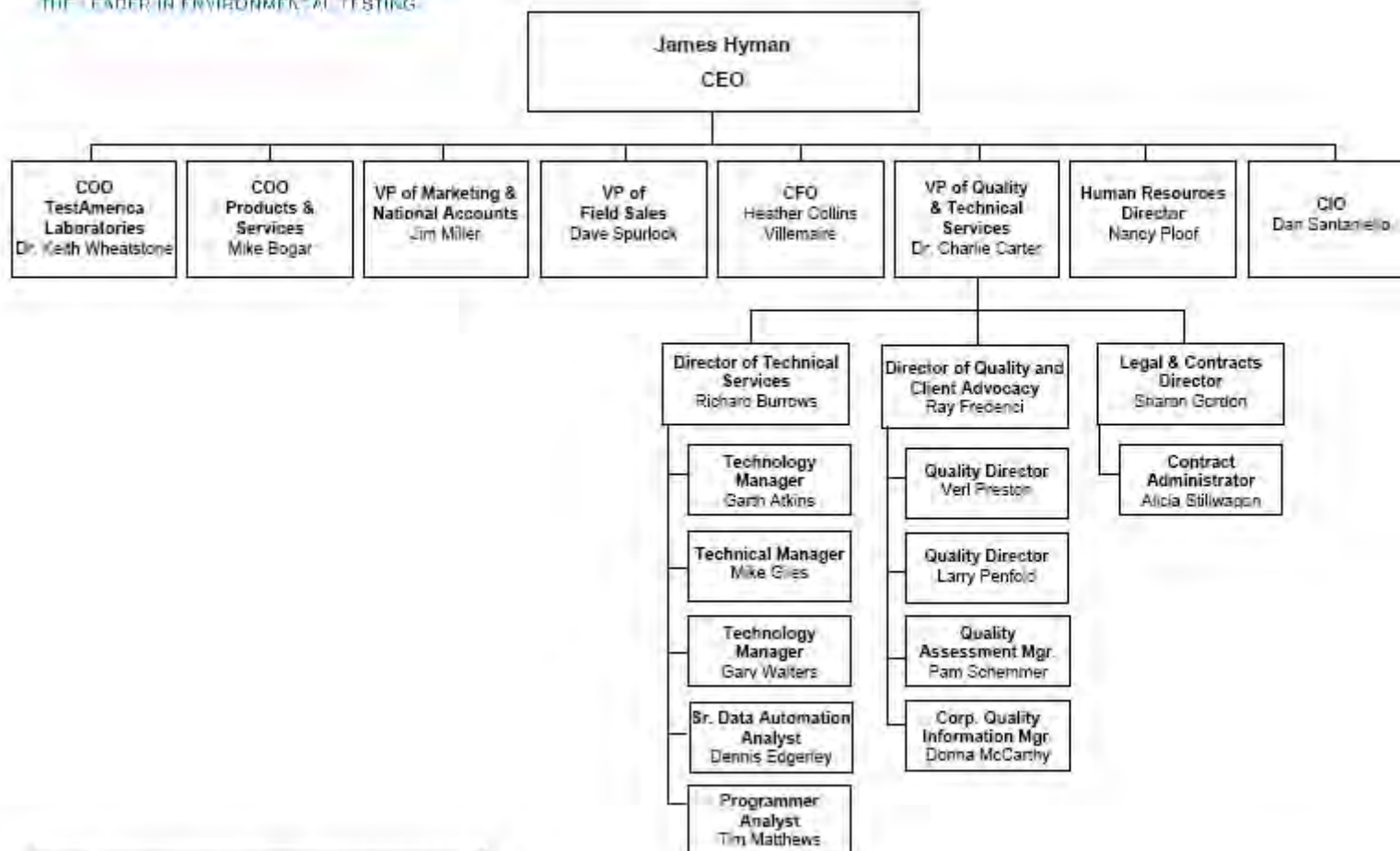
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying the QA manager of deficiencies in the quality system and ensuring corrective action is taken.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025. (where applicable)

#### 4.2 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Karla Buechler Laboratory Director	David J. Herbert Business Development Director
Douglas Weir Quality Manager	Lisa Stafford Quality Assurance Scientist
Karla Buechler Technical Director	Eric Redman Technical Director Robert Hrabak Environmental Operations Manager
Robert Hrabak Environmental Operations Manager	Steve Rogers Department Manager Kirby Garrett Department Manager
David Allameh Adv. Tech. Operations Manager	Robert Hrabak Environmental Operations Manager
David Herbert Business Development Manager	Eric Redman Technical Director
David Herbert Customer Service Manager	Jill Kellmann Program Management Department Manager
Joseph Schairer EHS Coordinator	Richard Kester Hazardous Materials Specialist

Figure 4-1. Corporate and Laboratory Organization Charts

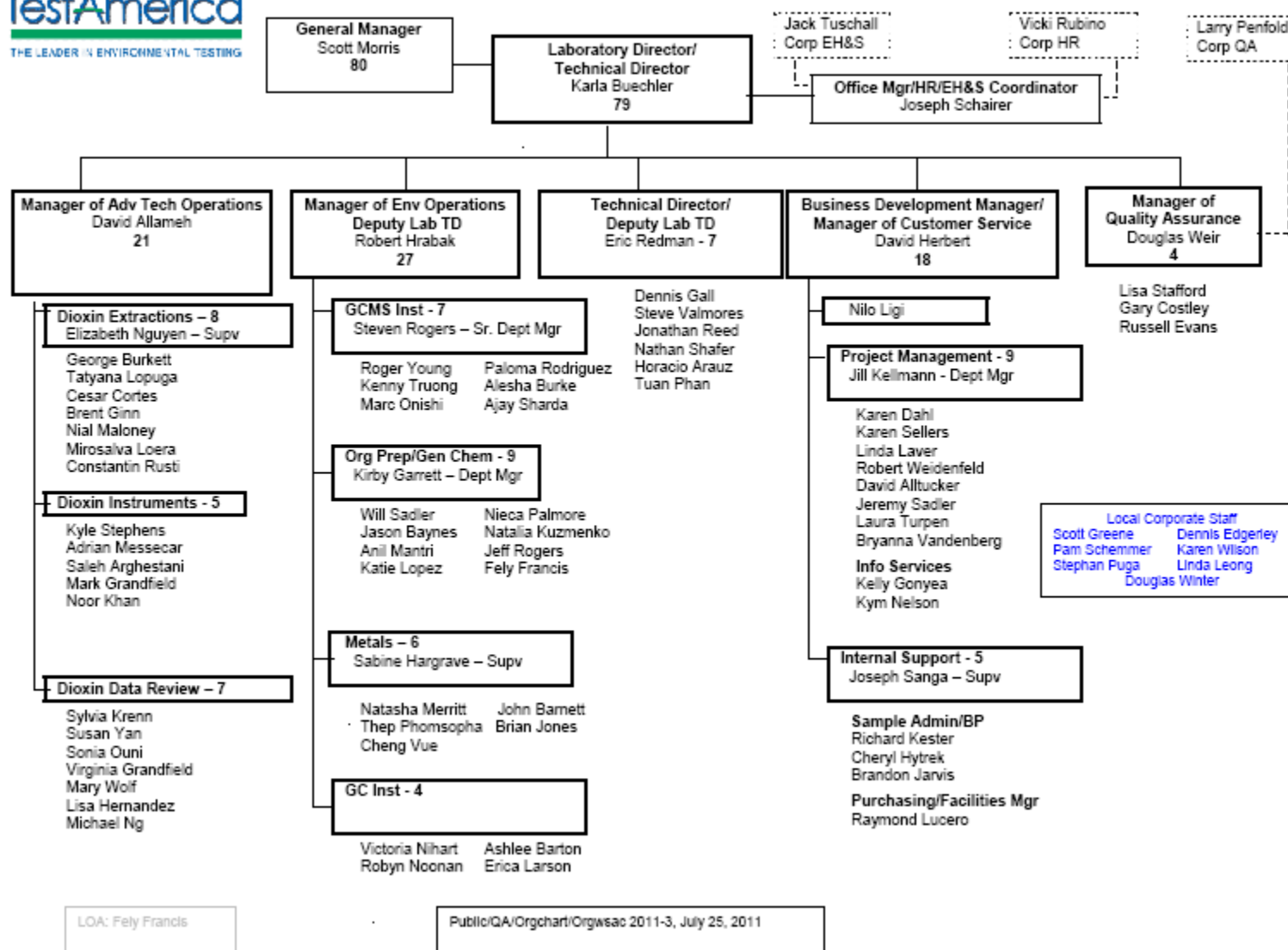


Note: QA Managers and Safety Coordinators in all laboratories and facilities have a dotted line reporting relationship to Corporate QA and EHS.

May 2011



## West Sacramento Laboratory Organization





## **SECTION 5. QUALITY SYSTEM**

### **5.1 Quality Policy Statement**

It is TestAmerica's Policy to:

- ❖ Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

### **5.2 Ethics and Data Integrity**

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (Corporate SOP No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.

- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

### **5.3 Quality System Documentation**

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

#### **5.3.1 Order of Precedence**

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

## **5.4 QA/QC Objectives for the Measurement of Data**

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term “*analytical quality control*”. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

### **5.4.1 Precision**

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

### **5.4.2 Accuracy**

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

#### **5.4.3      Representativeness**

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

#### **5.4.4      Comparability**

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

#### **5.4.5      Completeness**

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

#### **5.4.6      Selectivity**

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

#### **5.4.7      Sensitivity**

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

#### **5.5            Criteria for Quality Indicators**

The laboratory maintains a Reference Data Summary from the LIMS that summarizes the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in "Quality Control Program" Policy WS-PQA-003 and Section 24.

#### **5.6            Statistical Quality Control**

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Quality Control Program" Policy WS-PQA-003 and Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

#### **5.6.1      QC Charts**

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. Control charts are generated according to laboratory SOP No. WS-PQA-003, "Quality Control Program".

#### **5.7          Quality System Metrics**

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

## **SECTION 6. DOCUMENT CONTROL**

### **6.1 Overview**

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

### **6.2 Document Approval and Issue**

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department (or list other mgmt. if applicable). In order to develop a new document, a manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their

distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

### **6.3 Procedures for Document Control Policy**

For changes to the QA Manual, refer to SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the QA share on the local server for the applicable revision, and are accessible using the laboratory's Intranet.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP and SOP No. WS-QA-0021, Preparation and Management of Standard Operating Procedures". The SOPs identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office. There is a table of contents. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

### **6.4 Obsolete Documents**

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. WS-QA-0021, Preparation and Management of Standard Operating Procedures.



## **SECTION 7. SERVICE TO THE CLIENT**

### **7.1 Overview**

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

## **7.2 Review Sequence and Key Personnel**

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. The PM will also get approval by the Laboratory Director to commit to delivery schedules that are shorter than the published standard TATs. The Laboratory Director updates these TATs on a routine basis, and it is the responsibility of CSMs and PMs to review them prior to making commitments for the laboratory.

It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

If the project is an air, drinking water, or high resolution opportunity, a message describing the opportunity will be immediately sent to the appropriate specialty market distribution list.

New opportunities with an estimated value greater than \$100K are passed to the laboratory CSM or BDM, and a message regarding the project details is immediately forwarded to the Large Opportunity Tracking (LOT) distribution list. Specialty market distribution will be included in this notification as appropriate, as well as the associated sales person.

The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- The Customer Service Manager
- The Business Development Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Legal Contracts Director, Account Executive, Proposal Coordinator or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts, as does the local Business Development Manager.

### **7.3 Documentation**

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Contract negotiations and finalization is the responsibility of the Business Development Manager. These records are archived by client and project in a restricted network folder accessible to laboratory department managers, project managers, and senior managers.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. Each Laboratory Project Manager keeps a phone log of conversations with the client. In addition, all conversations involving notification of important information, or actions directed by the client are documented with a follow up e-mail and archived in the contracts folder or the SDG documentation and case narrative. Instances include change in scope, alterations to the requests listed on a chain of custody, directions to proceed in the event of a non-conformance, and any other conversation that changes the direction of a COC or contract.

#### **7.3.1 Project-Specific Quality Planning**

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Quality Assurance Project Plans, if submitted by the client, will be evaluated per policy WS-PQA-0018.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition,

project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are updated to the QAS and introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

#### **7.4      Special Services**

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

**Note:** ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

#### **7.5      Client Communication**

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Any member of the laboratory's senior staff, or any of the laboratory's identified technical experts is available to discuss any technical questions or concerns that the client may have.

## **7.6      Reporting**

The laboratory works with our clients to produce any special communication reports required by the contract.

## **7.7      Client Surveys**

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

## **SECTION 8. SUBCONTRACTING OF TESTS**

### **8.1 Overview**

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOP’s on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Account Executives (AE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

**Note:** In addition to the client, some regulating agencies (e.g, USDA) or contracts (e.g, certain USACE projects) may require notification prior to placing such work. Documentation of approval is stored electronically in the quote folder within SACSALES share on a local laboratory server.

### **8.2 Qualifying and Monitoring Subcontractors**

Whenever a PM or Customer Service Manager becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site.

Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable, (e.g., on the subcontractors TNI, A2LA accreditation or State Certification).

- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- TNI or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, PMS or CSMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

**8.2.1** Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site and notify the finance group for JD Edwards.

**8.2.2** The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

**8.2.3** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.



- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Laboratory Directors, QA Managers and Sales Personnel.

### **8.3      Oversight and Reporting**

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or CSM) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is stored electronically in the quote folder within the SACSALES share on a local laboratory server. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client CoCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client CoCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples. Results submitted by a network work-sharing laboratory on the same LIMS will be designated in the case narrative.

**Note:** The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods



and samples. The final report must include a copy of the completed COC for all work sharing reports.

#### **8.4      Contingency Planning**

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

**Figure 8-1.**

**Example 1 - Subcontracted Sample Form – form**

**Date/Time:** \_\_\_\_\_

**Subcontracted Laboratory Information:**

- Subcontractor's Name: \_\_\_\_\_
- Subcontractor Point of Contact: \_\_\_\_\_
- Subcontractor's Address: \_\_\_\_\_
- Subcontractor's Phone: \_\_\_\_\_
- Analyte/Method: \_\_\_\_\_
- Certified for State of Origin: \_\_\_\_\_
- TNI Certified: Yes \_\_\_\_\_ No \_\_\_\_\_
- **USDA Permit ( \_\_ Domestic \_\_ Foreign)** Yes \_\_\_\_\_ No \_\_\_\_\_
- A2LA (or ISO 17025) Certified: Yes \_\_\_\_\_ No \_\_\_\_\_
- CLP-like Required:  
(Full doc required) Yes \_\_\_\_\_ No \_\_\_\_\_
- Requested Sample Due Date:  
(Must be put on COC) \_\_\_\_\_
- **Client POC Approval on-file to  
Subcontract Samples to Sub Laboratory:** Yes \_\_\_\_\_ No \_\_\_\_\_

**Project Manager:** \_\_\_\_\_

**Laboratory Sample # Range:** \_\_\_\_\_  
(Only of Subcontracted Samples)

**Laboratory Project Number (Billing Control #):** \_\_\_\_\_

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

**PM Signature** \_\_\_\_\_ **Date** \_\_\_\_\_

**Example 1 - Subcontracted Sample Form – Within TestAmerica**

<b>TestAmerica</b>	<b>Work Sharing Agreement</b> (Include with every sample shipment)		<b>Exporting Lab</b> <b>Project Name</b>		<b>TAL Chicago</b>	
<b>Import Lab Information</b>			<b>Export Lab Information</b>			
Lab Name: TAL Chicago			PM Contact Name:			
PM Contact Name:			Backup Contact Name:			
Backup Contact Name:			Agreement Date:			
<b>Pricing Information</b>			<b>Project Information</b>			
QA/QC (i.e. MS/MSD) Billable? No			Client Company Name:			
Raw Data Surcharge %			Date First Samples to Arrive:			
EDD Surcharge %			Est. Duration of Sampling Event:			
TAT Surcharge %						
Penalty Terms: None						
Other Charges Not in Unit Price? (i.e. containers, Level VIII, shipping, bottles) None			Quote or Contract Reference ID:			
<b>Project Details</b>						
Non-Standard Work Product: No						
Quality Assurance Plan: No						
List Certifications Required:						
Analyte/Compd. List with RLs Attached: No						
Results Dry-Weight Corrected: No						
Special Method Holding Times: None						
Internal Chain of Custody Required: No						
Known Hazards/High Analyte Level: No						
Saturday/Special Delivery Options: None						
Special Instructions: None						
Reporting Limit Value Convention: Report to MDL with "J" Values up to "Non-Detect" Presentation Convention U						
<b>Deliverable Requirements</b>			Transmittal medium:	Format Column:	Date Due to Export Lab:	<b>Import and Export Lab Agreement</b>  Import lab must acknowledge receipt of Agreement and samples via E-Mail
Preliminary Report: No						
Final Report: No						
EDD: No			NA	Type Format Name:		
Total Access Reporting: No			NA	NA		
Custom Forms: No			NA	Type Format Name:		
<b>"Other" Deliverable Definitions or Notes</b>						
<b>Analysis</b>	<b>Method</b>	<b>Matrix</b>	<b># of Samples</b>	<b>Import Lab's Unit Price</b>	<b>Unit Price w/Surcharges</b>	<b>Extended Price</b>
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					\$ -	\$ -
					<b>Approximate Total Project Value</b>	
					\$ -	

Work Instruction No. CA-WI-010/B-10/08

## **SECTION 9. PURCHASING SERVICES AND SUPPLIES**

### **9.1 Overview**

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

### **9.2 Glassware**

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

### **9.3 Reagents, Standards & Supplies**

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

#### **9.3.1 Purchasing**

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Many items used routinely are pre-qualified and placed into the on-site consignment system.

For items not available from the consignment system, or items that are not used routinely, an order is placed in the JDE ordering system. Only personnel trained in the ordering program JDE may place orders using the program. All relevant information, including quantity, must be entered. Only approved vendors may be used. A vendor must be approved by corporate to be

on the approved vendor list in JDE. The Laboratory Director or designee approves all orders placed in JDE.

### **9.3.2      Receiving**

It is the responsibility of the purchasing manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials here received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

### **9.3.3      Specifications**

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOP's expiration date unless 'verified'. See laboratory SOP No. WS-QA-0017, "Standards and Reagent Preparation and Quality Control Check Procedures", for standard verification procedures. )

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning every other day. For single tanks in use at the bench, the minimum total pressure must be 500 psig or the tank must be replaced. To prevent a tank from going to dryness, close observation of the tank gauge must take place as pressure decreases towards 500 psig, or the tank must be replaced. For the automated "tank farm" in use through most of the laboratory, the minimum total pressure at which the system switches to the next bank of tanks is 250 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1-  $\mu\text{mho/cm}$  (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified “clean” by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard. See laboratory SOP No. WS-QA-0017, “Standards and Reagent Preparation and Quality Control Check Procedures”, for standard QC procedures.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer’s certification and traceability statements are maintained in files or binders in each laboratory section. Certificates of analysis are also scanned and stored electronically. These records include date of receipt, lot number (when applicable), and expiration date (when applicable).

#### **9.3.4      Storage**

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

#### **9.4          Purchase of Equipment / Instruments / Software**

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager/Director and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica’s Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer’s operation manual is retained at the bench and inventoried in the master document list.

#### **9.5          Services**

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is

determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

## **9.6 Suppliers**

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

### **9.6.1 New Vendor Procedure**

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technology Director are consulted with vendor and product selection that have an impact on quality.

## **SECTION 10. COMPLAINTS**

### **10.1 Overview**

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following laboratory policy WS-PQA-013, Procedure to Address Customer Complaints.

### **10.2 External Complaints**

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to laboratory policy WS-PQA-013, Procedure to Address Customer Complaints.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.



### **10.3      Internal Complaints**

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

### **10.4      Management Review**

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

## **SECTION 11. CONTROL OF NON-CONFORMING WORK**

### **11.1 Overview**

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

### **11.2 Responsibilities and Authorities**

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CW-L-S-002) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc.. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be

documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, the COO, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

### **11.3      Evaluation of Significance and Actions Taken**

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

### **11.4      Prevention of NonConforming Work**

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, (or add the lab's schedule; i.e., monthly, weekly) the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

### **11.5      Method Suspension / Restriction (Stop Work Procedures)**

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager/Director, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

## **SECTION 12. CORRECTIVE ACTION**

### **12.1 Overview**

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

### **12.2 General**

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc..

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

**12.2.1 Non-Conformance Memo (NCM)** - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

**12.2.2 Corrective Action Report (CAR)** - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints

- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and Safety violations

This will provide background documentation to enable root cause analysis and preventive action.

### **12.3 Closed Loop Corrective Action Process**

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

#### **12.3.1 Cause Analysis**

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Laboratory SOP No. WS-QA-0023, Nonconformance and Corrective Action System, provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

#### **12.3.2 Selection and Implementation of Corrective Actions**

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

#### **12.3.3 Root Cause Analysis**

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the

likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

#### **12.3.4 Monitoring of the Corrective Actions**

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and are periodically reviewed to ensure that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

#### **12.3.5 Follow-up Audits**

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

#### **12.4 Technical Corrective Actions**

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred



(refer to Section 11). The documentation of these procedures is through the use of an NCM or CAR.

Laboratory SOP No. WS-QA-0023, "Nonconformance and Corrective Action System" includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Laboratory SOP No. WS-QA-0023, "Nonconformance and Corrective Action System" provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The SOP also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

## **12.5      Basic Corrections**

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.



**Figure 12-1.**  
**Example - Corrective Action Report**

		<b>West Sacramento Corrective Action Report</b>	
<b>Title:</b> <Enter Title Here>			
<b>Reference:</b> <tracking information>			
<b>Initiated by:</b>		<b>Date:</b>	
<b>Responsible Party:</b>		<b>Date:</b> <date report submitted>	
<b>Description of Problem:</b> [enter text to briefly explain how the problem was discovered, who discovered it and when, and what work, if any, is affected]			
<b>Investigation Planned or Completed:</b> [enter text to briefly what was examined to determine the extent of the problem, when the investigation was conducted, what was the proximate cause(s), and what were the root causes. The key is to demonstrate that the investigation is comprehensive]			
<b>Root Cause Analysis</b> [True root cause analysis should involve multiple layers of questioning] <i>Examples:</i> <ul style="list-style-type: none"><li>- Why did this problem occur?</li><li>- What weaknesses are indicated by this problem?</li><li>- What Quality/ Systems mechanisms are in place that should have prevented this problem from occurring?</li><li>- Is this issue acute or chronic?</li><li>- Are changes needed to existing SOPs to correct this problem and prevent its recurrence?</li><li>- Are other departments affected by this issue?</li></ul>			
<b>Corrective Action Plan</b> [Based on the Root Cause Analysis outlined above, what action items need to be completed to correct this deficiency, and prevent its recurrence?] <i>Examples:</i> <ul style="list-style-type: none"><li>- Identify impacted lots</li><li>- Revise results/reports</li><li>- Initiate formal Data Recall</li><li>- Revise SOP</li><li>- Re-train staff</li></ul>			
<b>QA Monitoring of Corrective Action Status</b> [If an anomalous or isolated event, and no further action required, this section may be omitted. Otherwise, note the need for a routine follow-up assessment and the associated details (responsible party, due date, documentation necessary), or the need to add to the internal audit checklist for reassessment at a later date.]			
<b>Closed by:</b>		<b>Date:</b>	
Douglas Weir QA Manager			

**Table 12-1. Example – General Corrective Action Procedures**

<b>QC Activity (Individual Responsible for Initiation/Assessment)</b>	<b>Acceptance Criteria</b>	<b>Recommended Corrective Action</b>
Initial Instrument Blank  (Analyst)	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards  (Analyst, Technical Manager(s))	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source)  (Analyst, Technical Manager(s))	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards  (Analyst, Data Reviewer)	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD)  (Analyst, Data Reviewer)	- % Recovery within limits documented in the LIMS or Project QAPP.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS)  (Analyst, Data Reviewer)	- % Recovery within limits documented in the LIMS or Project QAPP.	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes.  <b>Note:</b> If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates  (Analyst, Data Reviewer)	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB)  (Analyst, Data Reviewer)	< Reporting Limit <sup>1</sup>	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples  (QA Manager, Technical Manager(s))	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits  (QA Manager, Technical Manager(s), Laboratory Director)	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Reporting / Calculation Errors  (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002.
Client Complaints  (Project Managers, Lab Director/Manager, Sales and Marketing)	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example)  (QA Manager, Lab Director/Manager, Technical Manager(s))	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation  (Safety Officer, Lab Director/Manager, Technical Manager(s))	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

**Note:**

1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants as defined in policy WS-PQA-003 provided they appear in similar levels in the reagent blank and samples. The ubiquitous contaminants include: methylene chloride, toluene, acetone, 2-butanone, phthalates and octachlorodibenzodioxin. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit

## **SECTION 13. PREVENTIVE ACTION / IMPROVEMENT**

### **13.1 Overview**

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluation of proficiency testing (PT) performance, data analysis & review processing operations, client complaints, staff observation, etc..

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc.. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

**13.1.1** The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

**13.1.2** Any Preventive Actions undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

## **13.2      Management of Change**

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in WS-QA-0050, Management of Change Procedures.

## SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

### 14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department in a database or in specific folders on the QA share on the local server, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Department Managers.

**Table 14-1. Record Index<sup>1</sup>**

	<u>Record Types<sup>1</sup>:</u>	<u>Retention Time:</u>
<b>Technical Records</b>	<ul style="list-style-type: none"> <li>- Raw Data</li> <li>- Logbooks<sup>2</sup></li> <li>- Standards</li> <li>- Certificates</li> <li>- Analytical Records</li> <li>- MDLs/IDLs/DOCs</li> <li>- Lab Reports</li> </ul>	5 Years from analytical report issue*
<b>Official Documents</b>	<ul style="list-style-type: none"> <li>- Quality Assurance Manual (QAM)</li> <li>- Work Instructions</li> <li>- Policies</li> <li>- SOPs</li> <li>- Policy Memorandums</li> <li>- Manuals</li> </ul>	5 Years from document retirement date*
<b>QA Records</b>	<ul style="list-style-type: none"> <li>- Internal &amp; External Audits/Responses</li> <li>- Certifications</li> <li>- Corrective/Preventive Actions</li> <li>- Management Reviews</li> <li>- Method &amp; Software Validation / Verification Data</li> <li>- Data Investigation</li> </ul>	5 Years from archival*  <u>Data Investigation:</u> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
<b>Project Records</b>	<ul style="list-style-type: none"> <li>- Sample Receipt &amp; COC Documentation</li> <li>- Contracts and Amendments</li> <li>- Correspondence</li> <li>- QAPP</li> <li>- SAP</li> <li>- Telephone Logbooks</li> <li>- Lab Reports</li> </ul>	5 Years from analytical report issue*



	<b><u>Record Types</u></b> <sup>1</sup> :	<b><u>Retention Time:</u></b>
<b>Administrative Records</b>	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records (Add Permits?)	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

<sup>1</sup> Record Types encompass hardcopy and electronic records.

<sup>2</sup> Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

\* Exceptions listed in Table 14-2.

**14.1.1** All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or the Iron Mountain data storage facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log. Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records before 09/09/2009 were maintained on-site at the laboratory for at least 1 month after their generation and moved offsite for the remainder of the required storage time. Records generated after 09/09/2009 are maintained on-site at the laboratory for at least 1 month after their generation and then scanned into PDF files. The electronic files are stored on-site and backed up daily offsite. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

#### **14.1.2 Programs with Longer Retention Requirements**

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.



**Table 14-2. Example: Special Record Retention Requirements**

<b>Program</b>	<b><sup>1</sup>Retention Requirement</b>
Drinking Water – All States	5 years (project records) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

<sup>1</sup>Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

**14.1.3** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 and WS-PQA-017 for more information.

**14.1.4** The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set.) Refer to SOP WS-QA-0009, Document Archiving. Instrument data is stored by project, except for inorganics and calibration data. Inorganics and calibration data is stored sequentially by instrument as appropriate. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP WS-QA-0009.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

## **14.2      Technical and Analytical Records**

**14.2.1**      The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

**14.2.2**      Observations, data and calculations are recorded real-time and are identifiable to the specific task.

**14.2.3**      Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72)

hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook, on a benchsheet or in the LIMS.

- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

#### **14.3      Laboratory Support Activities**

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and

- results of data review, verification, and crosschecking procedures

#### **14.3.1 Sample Handling Records**

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

#### **14.4 Administrative Records**

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

#### **14.5 Records Management, Storage and Disposal**

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in a logbook or using the Veritas Electronic Standards Logbook. Records are considered archived when noted as such in the records management system (a.k.a., document control.)

##### **14.5.1 Transfer of Ownership**

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in

cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

#### **14.5.2     Records Disposal**

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

## SECTION 15. AUDITS

### 15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

**Table 15-1. Types of Internal Audits and Frequency**

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-004)	Methods Audits Frequency: 100% of methods annually (DoD Labs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

#### 15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

#### **15.1.2     QA Technical Audits**

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

#### **15.1.3     SOP Method Compliance**

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

#### **15.1.4     Special Audits**

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

#### **15.1.5     Performance Testing**

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Soil, Water Supply, Water Pollution, Air, and round-robin studies for sediments and biological materials.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

#### **15.2       External Audits**

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are



responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

### **15.2.1 Confidential Business Information (CBI) Considerations**

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

### **15.3 Audit Findings**

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.



## **SECTION 16. MANAGEMENT REVIEWS**

### **16.1 Quality Assurance Report**

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

### **16.2 Annual Management Review**

The senior lab management team (Laboratory Director, Technical Managers, QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.

- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

### **16.3 Potential Integrity Related Managerial Reviews**

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

## **SECTION 17. PERSONNEL**

### **17.1 Overview**

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

### **17.2 Education and Experience Requirements for Technical Personnel**

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, Conductivity, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers – <b>General</b>	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry  An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – <b>Wet Chem</b> only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

### 17.3 **Training**

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP (WS-QA-0022, Employee Orientation and Training).

#### **17.4 Data Integrity and Ethics Training Program**

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

## **SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS**

### **18.1 Overview**

The laboratory is a 66,000 ft<sup>2</sup> secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

### **18.2 Environment**

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. In the event of a power outage, the laboratory can be equipped with a back up power supply for sample storage, as detailed in SOP No. WS-QA-0005, Temperature Monitoring and Corrective Action for Refrigerators and Freezers.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.



Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

### **18.3      Work Areas**

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

### **18.4      Floor Plan**

A floor plan can be found in Appendix 1.

### **18.5      Building Security**

Building keys and alarm codes are distributed to employees as necessary.

Employees wear photographic identification name cards while on the premises.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.



## **SECTION 19. TEST METHODS AND METHOD VALIDATION**

### **19.1 Overview**

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

### **19.2 Standard Operating Procedures (SOPS)**

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP WS-QA-0021 (Preparation and Management of Standard Operating Procedures).
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

### **19.3 Laboratory Methods Manual**

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

## 19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

### 19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)
- Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994
- NIOSH Manual of Analytical Methods, 4<sup>th</sup> ed., August 1994.
- Statement of Work for Inorganics & Organics Analysis, SOM, DLM, CBC, and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.

- Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup>/19<sup>th</sup>/20<sup>th</sup>/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.
- Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261
- Underground Storage Tanks Procedures Manual, State of Alaska Department of Environmental Conservation, Division of Spill Prevention and Response Contaminated Sites Program, November 7, 2002
- Tri-Regional Board Staff Recommendations for Preliminary Investigation and Evaluation of Underground Tank Sites, North Coast Regional Water Quality Control Board, San Francisco Bay Regional Water Quality Control Board and Central Valley Regional Water Quality Control Board, August 10, 1990
- Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997
- Compendium of Methods for the Determination of Air Pollutants in Indoor Air, (EPA 600/4-90-10, April 1990)
- Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, (EPA 625/R-96/010a, June 1999)
- Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources, Stationary Source Test Methods, Volume 3, California Air Resources Board

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

#### **19.4.2     Demonstration of Capability**

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC, Lab SOP # WS-QA-0022) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

#### **19.4.3     Initial Demonstration of Capability (IDOC) Procedures**

**19.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.

**19.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

**19.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

**19.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

**19.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

**19.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

**19.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

In accordance with Arizona Administrative Code R9-14-616.5f, documentation of each analyst's performance of proficiency testing, as applicable, will be maintained in the training record.

## **19.5      Laboratory Developed Methods and Non-Standard Methods**

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

## **19.6      Validation of Methods**

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

### **19.6.1      Method Validation and Verification Activities for All New Methods**

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

#### **19.6.1.1      Determination of Method Selectivity**

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

#### **19.6.1.2      Determination of Method Sensitivity**

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

#### **19.6.1.3      Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)**

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

#### **19.6.1.4      Determination of Interferences**

A determination that the method is free from interferences in a blank matrix is performed.



#### **19.6.1.5 Determination of Range**

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

#### **19.6.1.6 Determination of Accuracy and Precision**

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

#### **19.6.1.7 Documentation of Method**

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

#### **19.6.1.8 Continued Demonstration of Method Performance**

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

### **19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)**

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Alternatively, the MDL may be determined using a series (ideally 50-100) of method blanks for "uncensored" methods which always return a signal (i.e., ICP).

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. WS-QA-0006 for details on the laboratory's MDL process.

### **19.8 Instrument Detection Limits (IDL)**

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

### **19.9 Verification of Detection and Reporting Limits**

Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and no more than 4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. The analytes must be qualitatively identified or see SOP No. WS-QA-0006 for other options. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

For DoD ELAP certified methods: Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP WS-QA-0006, Method Detection Limits (MDL) and Instrument Detection Limits (IDL) for further details.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

For DoD ELAP certified methods: The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the



Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

#### **19.10 Retention Time Windows**

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

#### **19.11 Evaluation of Selectivity**

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

#### **19.12 Estimation of Uncertainty of Measurement**

**19.12.1** Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor  $k=2$ .

**19.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**19.12.3** The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent

recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**19.12.4** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/L, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/L, which could also be written as 1.0 +/- 0.5 mg/L.

**19.12.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

### **19.13      Sample Reanalysis Guidelines**

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within  $\pm 1$  reporting limit for samples  $\leq 5x$  the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Manager or Laboratory Director if unsure.

### **19.14      Control of Data**

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

#### **19.14.1      Computer and Electronic Data Related Requirements**

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP Nos. S-ITQ-005, "Quantlms/JDE user Profile Setup and Maintenance", and S-ITQ-007, "Software Testing, Validation and Verification." The laboratory is currently running Quantlms which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes DB2 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

**19.14.1.1 Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

**19.14.1.2 Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

**19.14.1.3 Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

#### **19.14.2 Data Reduction**

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and WS-PQA-011, Manual Integration Documentation Procedures.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff.

Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

- 19.14.2.1** All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.
- 19.14.2.2** In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter ( $\mu\text{g/L}$ ) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ( $\mu\text{g/kg}$ ) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%. Units are defined in each lab SOP.
- 19.14.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.
- 19.14.2.4** For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.
- 19.14.2.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

### **19.14.3      Logbook / Worksheet Use Guidelines**

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"ed out, signed and dated.

- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

#### **19.14.4      Review / Verification Procedures**

Review procedures are out lined in several SOPs (WS-PQA-003, "Quality Control Program", WS-PQA-012, "Technical Data Review Requirements", WS-PM-0004, "Final Report Assembly and Third Level Data Review") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (WS-PQA-0011, "Manual Integration Documentation and Practices"). The general review concepts are discussed below, more specific information can be found in the SOPs.

**19.14.4.1** The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

**19.14.4.2** The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add data qualifiers if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. One hundred percent of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

- 19.14.4.3** Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.
- 19.14.4.4** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.
- 19.14.4.5** As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.
- 19.14.4.6** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.
- 19.14.4.7** A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

#### **Manual Integrations**

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP No. WS-PQA-0011, entitled "Manual Integration Documentation and Practices.

- 19.14.4.8** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- 19.14.4.9** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission

of correct information) is against company principals and policy and is grounds for immediate termination.

**19.14.4.10** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

**19.14.4.11** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.



Figure 19-1. Example - Demonstration of Capability Documentation



### Demonstration of Capability Certification Statement

TestAmerica West Sacramento  
880 Riverside Parkway  
West Sacramento, CA 95605  
(916) 373-6500

Date: 28 June 2011  
Method: 1613B  
Matrix: Aqueous  
SOP: WS-ID C007 rev. 3.6

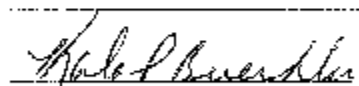
Analyst(s): M. GRANFIELD

We, the undersigned, CERTIFY that:

- 1: The analyst(s) identified above, using the cited test method, with the specifications in the cited SOP, which is in use at the facility for the analysis of samples under the TestAmerica West Sacramento Quality Assurance Manual, has met the Demonstration of Capability.
- 2: The test method was performed by the analyst(s) identified on this certification following the TestAmerica West Sacramento SOP.
- 3: A copy of the laboratory-specific SOP is available for all personnel on-site.
- 4: The data associated with the demonstration of capability are true, accurate, complete and self-explanatory ("). These data are attached to this certification statement.
- 5: All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/ Observations: Prep Chemists: B. GINN, M. LOERA.

Karla Buechler  
Technical Director

  
Technical Director Signature

7/15/11  
Date

Douglas Wei  
QA Manager

  
QA Manager Signature

07/15/11  
Date



TestAmerica

West Sacramento  
Water Proficiency Demonstration (DOC)

Note: Both averages are calculated based on the amount recovered, not the calculated recovery.  
RSD's are calculated based on the amount recovered, not the calculated recovery.

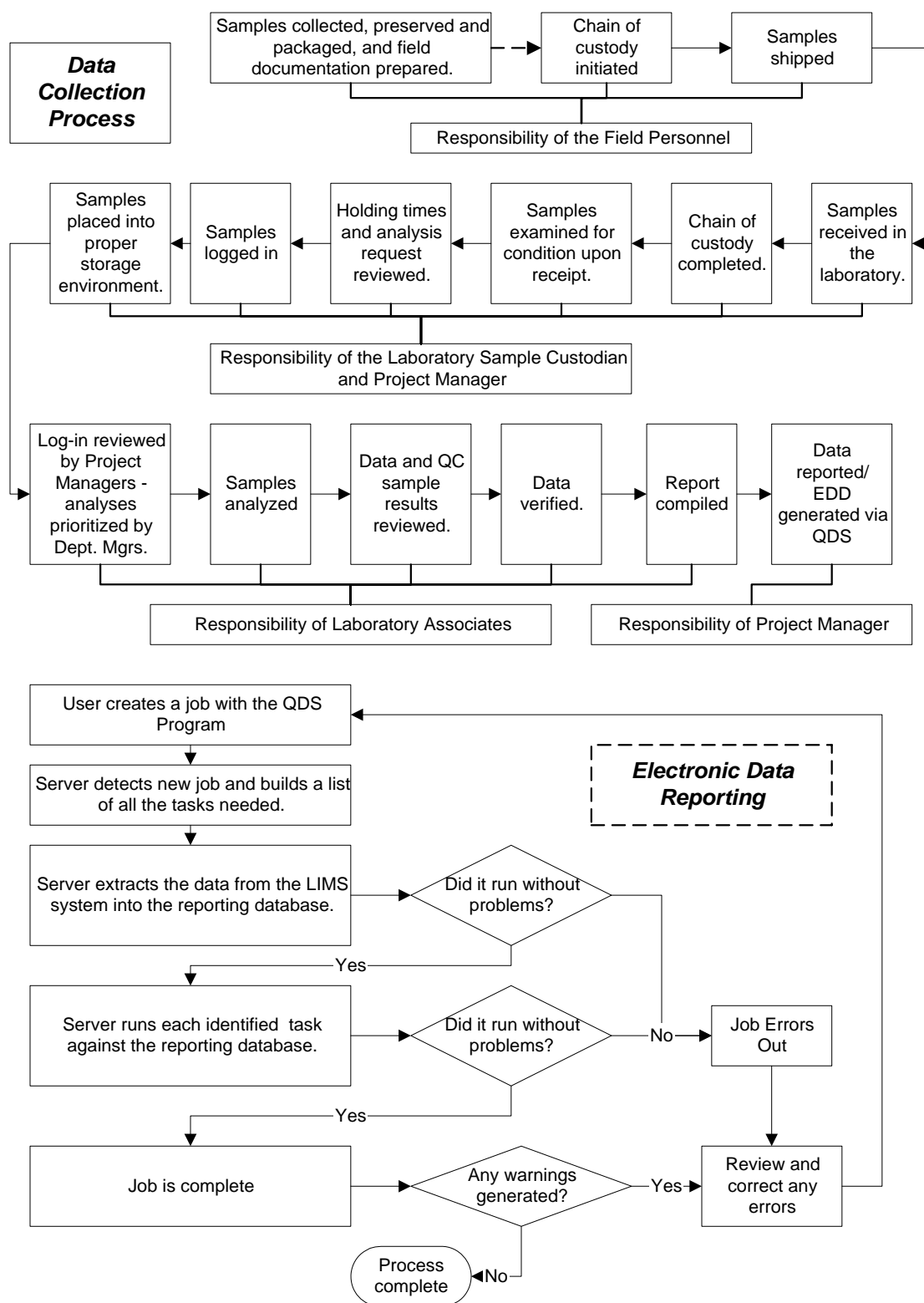
Data Completed:		6/2/11		Prep Analyst												
Method ID:		1613B		Analyst: Analyst: Mark Grandfield												
Method Description:		1613B		SCP (Include revision #) WS-IDP-0007 Rev. 1.3, WS-ID-0007 Rev. 3.5												
Prep Method:		Std. Pooled														
Analyte	Spike pg/L	Amount Recovered				Percent Recovery				AVE %	AVE pg/L	RSD %	Control Limits			
		LCS 1 pg/L	LCS 2 pg/L	LCS 3 pg/L	LCS 4 pg/L	LCS 1 %	LCS 2 %	LCS 3 %	LCS 4 %				Lower	Upper	RSD	
2,3,7,8-TCDF	200.000	234.235	209.470	215.380	124.810	117	105	108	97	107	212.484	7.8	75	158	50	ok
2,3,7,8-TCDF	200.000	215.380	211.573	214.690	138.639	110	106	107	94	107	219.819	8.0	67	158	50	ok
1,2,3,7,8-PeCDF	1000.000	1057.224	978.193	1077.813	1117.421	105	98	103	112	105	1057.428	5.4	60	154	50	ok
2,3,7,8-PeCDF	1000.000	1026.475	978.012	1064.704	1084.275	108	94	103	108	104	1044.178	4.9	80	150	50	ok
1,2,3,7,8-PeCDD	1000.000	1039.441	1008.994	1094.695	1155.304	103	101	99	111	104	1094.259	10.1	70	142	50	ok
1,2,3,4,7,8-HxCDF	1000.000	1112.400	1087.117	1075.470	1054.178	111	105	108	103	107	1075.032	3.1	72	134	50	ok
1,2,3,7,8-HxCDF	1000.000	1120.142	1057.908	1089.911	1059.491	112	105	109	107	106	1067.584	5.1	84	130	50	ok
2,3,4,6,7,8-HxCDF	1000.000	1112.312	1070.087	1091.694	1066.249	112	107	105	106	106	1074.084	9.2	70	155	50	ok
1,2,3,7,8-HxCDF	1000.000	1150.850	1094.104	1005.535	1067.179	113	102	104	108	107	1069.571	5.3	78	150	50	ok
1,2,3,4,7,8-HxCDD	1000.000	1088.002	1081.754	1075.496	1032.120	91	105	101	100	89	1094.008	7.7	70	154	50	ok
1,2,3,7,8-HxCDD	1000.000	1173.124	1072.897	1081.906	1111.875	118	102	108	111	101	1083.794	6.1	72	134	50	ok
2,3,7,8,9-HxCDD	1000.000	1049.111	1100.192	975.076	1163.703	103	113	87	117	102	1075.605	7.5	64	158	50	ok
2,3,7,8,9-HxCDD	1000.000	1025.111	1063.271	1048.314	1073.691	102	103	104	107	101	1037.823	2.9	82	122	50	ok
2,3,4,7,8,9-HpCDF	1000.000	1015.457	1064.458	1050.361	1053.692	101	107	106	105	105	1051.083	2.2	78	134	50	ok
1,2,3,4,6,7,8-HpCDD	1000.000	1073.033	1025.035	1058.704	1058.294	106	102	97	107	107	1073.035	1.5	70	146	50	ok
TCDF	2000.000	2298.850	2098.273	2134.771	2430.227	114	106	107	117	110	2137.885	4.8	66	170	50	ok
TCDD	2000.000	2270.841	2071.802	2021.654	2184.153	111	104	101	103	105	2124.740	1.4	78	144	50	ok
1,2,3,7,8-TCDF	2000.000	440.266	927.074	790.008	689.146	22	46	39	31	34	558.150	28.6	24	152	50	ok
1,2,3,7,8-TCDF	2000.000	427.604	920.957	661.199	571.590	21	44	30	28	29	548.180	32.2	20	175	50	ok
1,2,3,7,8-PeCDF	2000.000	454.547	1031.000	1095.604	572.204	22	62	31	28	34	660.890	36.0	21	182	50	ok
1,2,3,7,8-PeCDF	2000.000	532.810	1096.358	691.136	591.711	27	53	35	32	37	730.173	34.8	19	202	50	ok
1,2,3,7,8-PeCDD	2000.000	485.722	1185.555	684.779	541.701	23	53	35	27	36	722.511	44.2	21	227	50	ok
1,2,3,7,8-PeCDD	2000.000	711.273	1081.340	800.134	722.770	35	48	40	36	40	801.674	16.2	19	202	50	ok
1,2,3,4,7,8-HxCDF	2000.000	610.048	954.590	725.042	787.248	30	45	38	40	40	783.255	19.2	21	178	50	ok
1,2,3,4,7,8-HxCDF	2000.000	672.928	1007.560	722.546	848.149	34	51	39	42	42	825.292	20.8	22	172	50	ok
1,2,3,4,7,8-HxCDF	2000.000	741.498	1127.089	778.708	818.284	35	55	39	41	41	855.099	21.9	17	305	50	ok
1,2,3,4,7,8-HxCDF	2000.000	815.355	1058.627	1043.958	657.324	40	48	40	33	42	832.026	15.2	21	178	50	ok
1,2,3,7,8,9-HxCDF	2000.000	678.485	1168.171	871.548	732.752	33	56	34	37	41	754.511	31.7	20	193	50	ok
1,2,3,7,8,9-HxCDF	2000.000	738.116	1195.903	691.095	654.015	37	60	37	35	41	825.355	23.0	19	178	50	ok
1,2,3,4,6,7,8-HpCDF	2000.000	758.263	1179.015	858.478	675.481	38	58	32	34	40	824.088	23.0	20	195	50	ok
1,2,3,4,7,8-HpCDD	2000.000	651.857	1144.846	687.641	620.501	34	57	34	30	39	791.656	31.4	20	186	50	ok
1,2,3,4,7,8-HpCDD	2000.000	1277.571	2149.613	1436.025	1575.553	52	54	38	35	40	1638.267	25.7	13	180	50	ok

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Page 1 of 1

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Version 5/20/2010

**Figure 19-2. Example: Work Flow**



## **SECTION 20. EQUIPMENT and CALIBRATIONS**

### **20.1 Overview**

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

### **20.2 Preventive Maintenance**

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be / are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on 'date' was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

### **20.3      Support Equipment**

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

#### **20.3.1      Weights and Balances**

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. See SOP No. WS-QA-0041, "Calibration and Calibration Check of Balances" for more details.

### **20.3.2 pH, Conductivity, and Turbidity Meters**

The pH meters used in the laboratory are accurate to  $\pm 0.1$  pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

### **20.3.3 Thermometers**

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The digital NIST thermometer is recalibrated every five years (choose based on thermometer type or insert your own frequency) (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP No. WS-QA-0016, "Thermometer Calibration."

### **20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators**

The temperatures of all refrigerator units and freezers used for sample storage are monitored 7 days a week; and each working day for units used for standard storage.

Ovens and waterbaths are monitored on days of use. Drying oven temperature must be recorded before and at the end of use. For example, an oven used for moisture determination must have its temperature recorded at the start and end of the drying process. Temperature must be  $\pm 5\%$  of set temperature for DoD work.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between  $> 0^{\circ}\text{C}$  and  $\leq 6^{\circ}\text{C}$ .

Specific temperature settings/ranges for other refrigerators, ovens, and waterbaths can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

### **20.3.5 Autopipettors, Dilutors, and Syringes**

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. See SOP WS-QA-0004, "Maintenance and Calibration Check of Fixed and Adjustable Volume Autopipettors, Autodispensers and Volumetric Containers".

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

### **20.3.6 Autoclaves**

Autoclaves used for sample digestion are capable of maintaining conditions of 15 psi at  $120^{\circ}\text{C}$  for 15 minutes. The temperature of the autoclave is verified quarterly.

## **20.4 Instrument Calibrations**

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day. Further details regarding the calculations involved are present in SOP No. CA-Q-S-005, "Calibration Curves (General)."

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.



If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

**Note:** Instruments are calibrated initially and as needed after that and at least annually however, the annual requirement does not apply to Isotope Dilution methods.

#### **20.4.1 Calibration Standards**

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

##### **20.4.1.1 Calibration Verification**

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

**Note:** The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after every 10 samples or injections, including matrix or batch QC samples.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with unacceptable calibration verification may be fully useable under the following special conditions, and reported based upon discussion and approval of the client:



a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

#### **20.4.1.2 Verification of Linear and Non-Linear Calibrations**

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used. Detailed calculations for each fitting method can be found in CA-T-P-002.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

#### **20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis**

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data

system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

## **20.6      GC/MS Tuning**

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

**Table 20-1. Example: Instrumentation List<sup>1</sup>**

<b>Instrument Type</b>	<b>Number in Use</b>
Autoanalyzer	2
Autotitrator	1
Cold-Vapor Analyzers	2
GC/HRMS	8
GC/MS - Semivolatiles	6
GC/MS - Volatiles	5
GC/MS/MS	1
GC-ECD/ECD	7
GC-FID/FID	2
HPLC	5
HPLC/MS/MS	4
ICP	1
ICP/MS	2
Ion Chromatograph	3
Spectrometer	1
TOC Analyzer	1

**Table 20-2. Example: Schedule of Routine Maintenance**

INSTRUMENT	MAINTENANCE	FREQUENCY
APCI/ESI LC/MS/MS	Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.	As Needed
	Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.	Daily <sup>(2)</sup>
	Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable.	Semi-Annually
	Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.	Annually
HIGH PRESSURE LIQUID CHROMATOGRAPH(1)	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements. Rinse flow cell with 1N nitric acid if dirty flow cell. Change pump seals when flow becomes inconsistent. Backflush column if applicable. Change in-line filters for solvents.	As Needed
	Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse delivery lines to prevent contamination of the new solvent. Check gas supply if applicable. Flush with an appropriate solvent to remove all bubbles. Pre-filter all samples.	Daily <sup>(2)</sup>
	Change pump seals.	Every 6-9 Months

INSTRUMENT	MAINTENANCE	FREQUENCY
GAS CHROMATOGRAPH <sup>(1)</sup>	Replace septum. Clean injector port Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturers suggested maintenance schedule Replace fuse. Reactivate external carrier gas dryers. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents. Check inlets, septa.	As Needed
	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. ECD: perform wipe test.	Daily <sup>(2)</sup>
	ECD: perform wipe test.	Semi-Annually
PURGE AND TRAP SYSTEMS	Change trap. Check purge flow. Flush lines (after foaming sample). Periodic leak checks (when replace traps/spargers) Replace/condition traps and/or spargers (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), and clean or replace glassware/spargers. Bake trap as needed to correct for high background. Change trap whenever loss of sensitivity, or erratic response or failing resolution is observed. Purge & trap autosamplers: leak check system, clean sample lines, valves.	As Needed
	Bake out trap & analyze primers (as needed) prior to commencing analysis.	Daily <sup>(2)</sup>
GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETER <sup>(1)</sup>	Replace septum. Clean injector port. Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Replace injection port liner when front portion of capillary column is removed. Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed. Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	<p>Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.</p> <p>Replace filaments when both filaments burn out or performance indicates need for replacement.</p> <p>Check mass calibration (PFTBA or FC-43).</p> <p>Check ion source and analyzer (clean, replace parts as needed).</p> <p>Check vacuum, relays, gas pressures and flows.</p> <p>Change oil in the mechanical rough pump.</p> <p>Relubricate the turbomolecular pump-bearing wick.</p> <p>HP 7673 Autosampler: Replace syringe.</p>	
	<p>Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.</p> <p>Check temperatures of injector, detector.</p> <p>Verify temperature programs.</p> <p>Check inlets, septa.</p> <p>Check baseline level.</p> <p>Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.</p> <p>Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.</p> <p>Autosampler: fill wash bottle, dispose of waste bottle contents.</p>	Daily <sup>(2)</sup>
	<p>Replace the exhaust filters on the mechanical rough pump every 1-2 years.</p>	Annually
GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETER <sup>(1)</sup>	<p>Full Bake-Out.</p> <p>Change oil in rotary pump.</p> <p>Change oil in diffusion pump. Replace o-rings.</p> <p>Solvent rinse the flight tube.</p> <p>Clean the first field free region.</p> <p>Check detector voltages.</p> <p>Clean and dust connectors, etc on the outside of the instrument.</p> <p>Check the vacuum: <math>\sim 5 \times 10^{-7}</math> MBAR on both analyzer ion gauges, and <math>\sim 5 \times 10^{-6}</math> MBAR on the source, with no helium flowing.</p> <p>Check isolation valve for leaks, correct if needed.</p> <p>Check for thermal trip by taking the magnet to maximum current, and verify that the coolant flow is acceptable.</p> <p>Replace septum.</p> <p>Clean injector port.</p> <p>Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.</p> <p>Replace injection port liner when front portion of capillary column is removed.</p> <p>Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.</p> <p>Replace filaments when performance indicates need for replacement.</p>	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	Check resolution sensitivity. Check stability. Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily <sup>(2)</sup>
COLD VAPOR ATOMIC ABSORPTION (LEEMAN PS 200) <sup>(1)</sup>	Change pump tubing. Check/change Hg lamp. Clean optical cell. Change drying tube. Grease pump.	As Needed
	Check sample tip for clogs. Check drying tube. Check pump tubing/drain tubing. Check gas pressure. Check liquid/gas separator. Check tubing.	Daily <sup>(2)</sup>
INDUCTIVELY COUPLED ARGON PLASMA/MASS SPECTROMETRY (ICAP/MS) <sup>(1)</sup>	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics. Measure quartz torch for proper alignment when removed and cleaned. Clean spray chamber and nebulizer. Clean all filters and fans. Check chiller coolant level. Check and drain oil mist eliminator on roughing pumps.	As Needed
	Check sample waste container level. Check quartz torch condition. Check RF coil. Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing. Check condition of sampler and skimmer cones. Check oil level of roughing pumps.	Daily <sup>(2)</sup>
	Replace oil in roughing pumps.	Every 2-3 Months
ICP <sup>(1)</sup>	Check that argon feed pressure is 80-120 psi. Check that chiller coolant pressure is 45-80 psig, no leaks. Check purge and shear gasses. Nitrogen purge gas pressure 40-120 psig, compressed air shear gas pressure 80-120 psig. Check radial purge and axial windows for deposits. Check that nebulizer is not clogged. Check that capillary tubing is clean and in good condition. Check that peristaltic pump windings are secure. Check that exhaust vent is operational Check that torch, glassware, aerosol injector tube are clean.	Daily <sup>(2)</sup>
	Clean plasma torch assembly to remove accumulated deposits. Check RF coil. Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance.	Monthly or As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	Clean filters on back of power unit to remove dust. Replace when needed: peristaltic pump tubing. sample capillary tubing. autosampler sipper probe. Check performance with manganese. Check O-rings. Clean/lubricate pump rollers	
	Check chiller coolant filter. (may require more or less frequently)	Semi-Annually
	Notify manufacturer service engineer for scheduled preventive maintenance service.	Annually
ION CHROMATOGRAPH <sup>(1)</sup>	Clean micromembrane suppressor when decreases in sensitivity are observed. Check fuses when power problems occur. Change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated. De-gas pump head when flow is erratic. Check all air and liquid lines for discoloration and crimping, if indicated. Check/change bed supports guard and analytical columns, if indicated.	As Needed
	Check plumbing/leaks. Check eluent level. Check gases. Check pump pressure. Check conductivity meter.	Daily <sup>(2)</sup>
	Check pump heads for leaks. Check filter (inlet).	Weekly
	Change pump seals. Change injection valve. Clean conductivity cell. Check conductivity cell for calibration.	Annually
ALPKEM COLORIMETRIC AUTO ANALYZER <sup>(1)</sup>	Prepare fresh reagents. Replace tubing. (About every 100 hours of use)	As Needed
	Check detector. Make sure there are no trapped bubbles in detector cell. Check Valves Check peristaltic tubing. Check sampler.	Daily <sup>(2)</sup>
	Clean pump, and XYZ Sampler.	Weekly
	Lubricate pump roller.	Monthly
	Clean pump rollers with steel wool and lubricate.	Semi-Annually
SYSTEA COLORIMETRIC AUTO ANALYZER <sup>(1)</sup>	Prepare fresh reagents. Replace waste tubing. Replace probes. Replace lamp	As Needed
	Perform washes. Perform filters autozero. Check temperatures.	Daily <sup>(2)</sup>
CHEMICAL OXYGEN DEMAND (COD) REACTOR <sup>(1)</sup>	Electronics serviced.	As Needed
	Check temperature with NIST reference thermometer.	Annually



INSTRUMENT	MAINTENANCE	FREQUENCY
AUTO TITRATOR <sup>(1)</sup>	Electronics serviced.	As Needed
	Calibrate with check standards. Inspect electrodes daily, clean as needed. Inspect electrode proper levels of filling solutions daily, fill as needed. Clean probe, each use. Prime buret Check rinse water reservoir.	Daily <sup>(2)</sup> (When Used)
CONDUCTANCE METER <sup>(1)</sup>	Electronics serviced. Replace batteries	As Needed
SPECTROPHOTOMETER <sup>(1)</sup>	Replace lamp. Replace fuse.	As Needed
	Check instrument manual. Perform wavelength calibration. Replace lamp annually or when erratic response is observed.	Annually
PH METER <sup>(1)</sup>	Clean electrode. Refill reference electrode.	As Needed
	Inspect electrode. Verify electrodes are properly connected and filled. Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer.	Daily <sup>(2)</sup>
TOTAL ORGANIC CARBON ANALYZER (OI 1010 AND SOLIDS)	Check injection port septum after 50-200 runs. Perform leak test. Calibrate reagent pumps. Change sample loops. Adjust flow. Indicating drying tube. NDIR zero, after 100 hours of use. Sample pump, after 2000 hours for use. Digestion vessel/condensation chamber. Permeation tube, after 2000 hours of use. NDIR cell, after 2000 hours of use.	As Needed
	Check: Nitrogen supply, (oxygen supply for solids). Persulfate supply (1010 unit). Acid supply (1010 unit). Rinse water reservoir supply (1010 unit). IR millivolts for stability (after 30 min. warm-up).	Daily <sup>(2)</sup>
TURBIDIMETER <sup>(1)</sup>	Electronics serviced.	As Needed
	Clean instrument housing.	Monthly
DIGESTION BLOCK	Check temperature with NIST thermometer.	Annually
SONICATOR <sup>(1)</sup>	Replace probe tip. Disassemble and clean sonicator probe tips. Tune sonicator assembly (if recommended by manufacturer)	As Needed
	Inspect probe tips for inconsistencies (etching/pitting).	Daily <sup>(2)</sup> (When Used)
ANALYTICAL/TOP LOADING BALANCES <sup>(1)</sup>	Check using ASTM Class 3 weights once daily or before use. Clean pan and weighing compartment.	Daily <sup>(2)</sup>
REFRIGERATORS/WALK-IN COOLERS <sup>(1)</sup>	Manufacturer cleaning and calibration.	Annually
	Refrigerant system and electronics serviced.	As Needed
	Temperatures checked and logged.	Daily <sup>(2)</sup>

INSTRUMENT	MAINTENANCE	FREQUENCY
OVENS <sup>(1)</sup>	Electronics serviced.	As Needed
	Temperatures checked and logged.	Daily <sup>(2)</sup>
ZYMARK PE WORKSTATION	<p>Change O-rings whenever there are visible leaks or poor sealing on the SPE columns.</p> <p>Sample lines are clean after samples have been extracted by SPE with a program "Clean Sample Lines" with methanol followed by water. Occasionally for a more rigorous cleaning, or after a highly contaminated sample, a mixture of methanol/DCM at 50:50 may be used in place of methanol, follow by methanol, then water (never use acetone).</p> <p>Syringe pump may be primed using a program "Prime Solvent Lines" whenever air bubbles are suspected in the lines from running out of solvents and whenever solvents are changed.</p> <p>Syringe pump in good condition – replace if showing signs of wear or suspected of poor performance.</p> <p>Sample pumps may be re-calibrated whenever major repairs are performed, or whenever the pumps are suspected to be out of calibration. Follow manufacturer's procedure for re-calibrating the sample pumps. For method 8330, the pump loads 1050 mL of sample on the SPE. It should used up the whole sample bottle (quart bottles and 1-L bottles).</p>	As Needed
SONICATION WATER BATH <sup>(1)</sup>	<p>If the water bath is dirty, empty and refill with tap water. A couple drops of anti-bacterial solution may be added to inhibit the growth of bacteria in the water.</p> <p>The water level in the sonication batch should be about 1.2 to 1 inch from the top while in operation. Do not allow sonication batch to operate with water bath at lower levels.</p> <p>If the level is low, add more water, if the levels is too high, remove water to the proper level.</p>	As Needed

#### **Footnotes to Preventive Maintenance Tables**

- (1) Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.
- (2) Daily checks and verifications are performed prior to instrument startup and are not documented in maintenance logs unless problems are noted.

## **SECTION 21. MEASUREMENT TRACEABILITY**

### **21.1 Overview**

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

### **21.2 NIST-Traceable Weights and Thermometers**

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

The calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of  $k=2$  to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica West Sacramento contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are

achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica West Sacramento does not use a firm who subcontracts the work.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

### **21.3      Reference Standards / Materials**

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

#### **21.4      Documentation and Labeling of Standards, Reagents, and Reference Materials**

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.]

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the departments, and on the local server. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP No. WS-QA-0017, "Standards and Reagent Preparation and Quality Control Check Procedures".

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material (for 1613B dioxin/furan analyses the purity must be 98% or corrections must be made).

**21.4.1**      All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database or standards logbook.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)

- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically or in logbooks for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from the preparation logbook)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the MSDS section of OASIS.

**21.4.3** In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions.
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

## **SECTION 22. SAMPLING**

### **22.1 Overview**

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

### **22.2 Sampling Containers**

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

#### **22.2.1 Preservatives**

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

### **22.3 Definition of Holding Time**

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs and regulators, such as the Alaska Department of Environmental Conservation, which determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

### **22.4 Sampling Containers, Preservation Requirements, Holding Times**

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative.

As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

## **22.5      Sample Aliquots / Subsampling**

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located SOP Nos. WS-QA-0018, "Subsampling and Compositing of Samples (Method ASTM D 6323-98)" and WS-QA-0028, "Multi-Incremental Subsampling of Soils and Sediments".



**Table 22-1.**  
**Holding Times, Preservation and Container Requirements: Drinking Water (SDWA)**

PARAMETER	CONTAINER	PRESERVATION <sup>1,2</sup>		HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
		Temp. <sup>23</sup>	Chemical		
Asbestos	Plastic/Glass	4°C	None	48 hours <sup>5</sup>	1 L
Coliforms (Total and Fecal)	Plastic/Glass <sup>20</sup>	10°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	30 hours <sup>21</sup>	120 mL
Cyanide	Plastic/Glass	4°C	NaOH to pH >12 Ascorbic acid <sup>9</sup> or Sodium arsenite <sup>9</sup>	14 days	500 mL
Fluoride	Plastic/Glass	None	None	None	250 mL
Perchlorate (EPA 331.0)	Plastic/Glass <sup>20</sup>	4°C	None Filtered, 1/3 Headspace to minimize anaerobic conditions	28 days	250 mL
Heterotrophic Plate Count	Plastic/Glass <sup>20</sup>	10°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	8 hours (24 hours <sup>22</sup> )	120 mL
Mercury	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	28 days	250 mL
Metals <sup>4</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>24</sup>	6 months	250 mL
Nitrate	Plastic/Glass	4°C	None	48 hours <sup>6</sup>	250 mL
Nitrate-Nitrite	Plastic/Glass	None	H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	250 mL
Nitrite	Plastic/Glass	4°C	None	48 hours	250 mL
THMs Only	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>9</sup> HCl to pH <2 may also be used	14 days	3 X 40 mL
Volatile Organic Compounds	Glass <sup>8</sup>	4°C	HCl to pH <2 Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>9</sup> or Ascorbic acid <sup>9</sup>	14 days / 24 hrs <sup>25</sup>	3 X 40 mL
EDB, DBCP, 1,2,3- TCP (EPA 504.1)	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days <sup>11</sup>	3 X 40 mL
Organochlorine Pesticides/PCBs (EPA 505) <sup>10</sup>	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days <sup>11</sup>	3 X 40 mL

PARAMETER	CONTAINER	PRESERVATION <sup>1,2</sup>		HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
		Temp. <sup>23</sup>	Chemical		
Nitrogen and Phos. Pesticides (EPA 507)	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days <sup>12</sup>	1 L
Total PCBs (EPA 508A)	Glass-Amber <sup>8</sup>	4°C	None	14 days <sup>13</sup>	1 L
Pesticides and PCBs (EPA 508.1) <sup>14</sup>	Glass-Amber <sup>8</sup>	4°C	HCl to pH <2 Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>9</sup>	14 days <sup>13</sup>	1 L
Chlorinated Acids (EPA 515.1)	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days <sup>12</sup>	1 L
Nitrosamines (EPA 521)	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days <sup>12</sup>	1 L
Semivolatiles (EPA 525.2)	Glass-Amber <sup>8</sup>	4°C	HCl to pH <2 Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>9</sup>	14 days <sup>13</sup>	1 L
N-Methylcarbamoyloxamines and N-Methylcarbamates (EPA 531.1)	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , Monochloroacetic Acid buffer to pH<3	28 days	3 X 60 mL
Acetamide Herbicide Degradates (EPA 535)	Glass-Amber <sup>8</sup>	4°C	Ammonium Chloride	14 days <sup>12</sup>	250 mL
Glyphosate (EPA 547)	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days	3 X 60 mL
Endothall (EPA 548)	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	4°C	None	7 days <sup>15</sup>	1 L
Diquat/Parquat (EPA 549.1)	Glass-Amber <sup>8</sup> (Silanized or PVC amber)	4°C	H <sub>2</sub> SO <sub>4</sub> to PH <2 Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>9</sup>	7 days <sup>16</sup>	1 L
Chlorinated Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides/Herbicides (EPA 551)	Glass <sup>8</sup>	4°C	Phosphate Buffer and Ammonium Chloride <sup>19</sup>	14 days <sup>17</sup>	3 X 60 mL
Haloacetic Acids (EPA 552.1)	Glass-Amber <sup>8</sup>	4°C	Ammonium Chloride	28 days <sup>18</sup>	250 mL
2,3, 7, 8 TCDD	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	1 year	1 L

#### Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the

**Key to Table**

- preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
  4. All metals except Hg.
  5. Instructions for containers, preservation procedures and holding times as specified in Method 100.2 must be adhered to for all compliance analysis including those conducted with Method 100.1.
  6. If the sample is chlorinated, the holding time for an un-acidified sample kept at 4°C is extended to 14 days.
  7. Nitrate-Nitrite refers to a measurement of total nitrite.
  8. With Teflon lined septum.
  9. If chlorinated, add reagent prior to acidification (for Cyanide, add before NaOH).
  10. Heptachlor has a 7 day hold time.
  11. 14 days until extraction. 24 hours after extraction.
  12. 14 days until extraction. 28 days after extraction.
  13. 14 days until extraction. 30 days after extraction.
  14. For cyanazine, cool to 4°C only.
  15. 7 days until derivitization. 1 day after derivitization.
  16. 7 days until extraction. 21 days after extraction.
  17. 14 days until extraction. 14 days after extraction.
  18. 28 days until extraction. 48 hours after extraction.
  19. Sodium Sulfite may be used as a dechlorinating agent in some instances. Verify with laboratory prior to sampling.
  20. Sterilized. Plastic must be Polypropylene.
  21. 40 CFR part 141.74 regulations to avoid filtration or disinfection state 8 hours (DW compliance testing). Most facilities are using either disinfection or filtration so the 8 would not apply in most cases.
  22. 40 CFR part 141.74 regulations for Disinfection By-Product rule state 8 hours (DW compliance testing) where SM 9215 allows up to 24 hours if sample is stored between > 0 and ≤ 4° C.
  23. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to ≤ 6°C is acceptable.
  24. Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.
  25. Holding Time is 24 hours if pH adjustment is not performed.

**Table 22-2**  
**Holding Times, Preservation and Container Requirements: NPDES – Bacteria, Protozoa, Toxicity Tests**

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION <sup>2,3</sup>		HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
		Temp.	Chemical		
Total, Fecal, and E.coli Coliforms	Plastic/Glass	10°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>6</sup>	6 hours	100 mL
Fecal Streptococci	Plastic/Glass	10°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>6</sup>	6 hours	100 mL
Enterococci	Plastic/Glass	10°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>6</sup>	6 hours	100 mL
Cryptosporidium	LPDE Plastic	0-8°C	None	96 Hours	500 mL
Giardia	LPDE Plastic	0-8°C	None	96 Hours	500 mL
Toxicity – Acute/Chronic	Plastic/Glass	≤ 6°C <sup>5</sup>	None	36 Hours	2 L

**Key to Table**

1. Plastic should be Polypropylene or other sterilizable plastic.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. Samples must not be frozen. Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present, when samples arrive, it is necessary to measure the temperature of the samples and confirm that the ≤ 6°C temperature has not been exceeded.
6. Should only be used in the presence of residual chlorine.

**Table 22-3**  
**Holding Times, Preservation and Container Requirements: NPDES - Inorganic**

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION <sup>2,3</sup>		HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
		Temp <sup>14</sup>	Chemical		
Acidity	Plastic/Glass	≤ 6°C	None	14 days	100 mL
Alkalinity	Plastic/Glass	≤ 6°C	None	14 days	100 mL
Ammonia	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	400 mL
BOD 5 Day	Plastic/Glass	≤ 6°C	None	48 hours	1000 mL
Boron	Plastic <sup>5</sup>	None	HNO <sub>3</sub> to pH<2	6 months	200 mL
Bromide	Plastic/Glass	None	None	28 days	100 mL
CBOD 5 Day	Plastic/Glass	≤ 6°C	None	48 hours	1000 mL
COD	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	100 mL
Chloride	Plastic/Glass	None	None	28 days	50 mL
Chlorine, Residual	Plastic/Glass	None	None	15 min. <sup>6</sup>	200 mL
Color	Plastic/Glass	≤ 6°C	None	48 hours	50 mL
Cyanide –Total <sup>16, 17</sup>	Plastic/Glass	≤ 6°C	NaOH to pH >12, 0.6 g Ascorbic Acid <sup>7</sup>	14 days	100 mL
Cyanide – Amenable <sup>16, 17</sup>	Plastic/Glass	≤ 6°C	NaOH to pH >12, 0.6 g Ascorbic Acid <sup>7</sup>	14 days	100 mL
Fluoride	Plastic	None	None	28 days	300 mL
Hardness	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>8</sup>	6 months	100 mL
Hexavalent Chromium	Plastic/Glass	≤ 6°C	Ammonium sulfate buffer pH = 9.3 - 9.7	28 days / 24 hrs <sup>15</sup>	200 mL
Hydrogen Ion (pH)	Plastic/Glass	None	None	15 min. <sup>6</sup>	200 mL
Kjeldahl and organic Nitrogen	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	500 mL
Mercury <sup>11</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	28 days	200 mL
Metals <sup>9,10</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>18</sup>	6 months	200 mL
Nitrate	Plastic/Glass	≤ 6°C	None	48 hours	100 mL
Nitrate-Nitrite	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	100 mL
Nitrite	Plastic/Glass	≤ 6°C	None	48 hours	100 mL
Oil and Grease	Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> or HCl to pH <2	28 days	1 L

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION <sup>2,3</sup>		HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
		Temp <sup>14</sup> .	Chemical		
Organic Carbon (TOC)	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> or HCl to pH <2 <sup>12</sup>	28 days	250 mL
Orthophosphate	Plastic/Glass	≤ 6°C	Filter within 15 min.	48 hours	250 mL
Oxygen, Dissolved Probe	Glass <sup>13</sup>	None	None	15 min. <sup>6</sup>	200 mL
Oxygen, Winkler	Glass <sup>13</sup>	None	Fix on site and store in dark.	8 hours	300 mL
Phenols	Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	500 mL
Phosphorus, Elemental	Glass	≤ 6°C	None	48 hours	250 mL
Phosphorus, Total	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	250 mL
Residue, Total	Plastic/Glass	≤ 6°C	None	7 days	1 L
Residue, Filterable	Plastic/Glass	≤ 6°C	None	7 days	1 L
Residue, Non-Filterable	Plastic/Glass	≤ 6°C	None	7 days	1 L
Residue, Settleable	Plastic/Glass	≤ 6°C	None	48 hours	1 L
Residue, Volatile	Plastic/Glass	≤ 6°C	None	7 days	1 L
Silica	Plastic <sup>5</sup>	≤ 6°C	None	28 days	250 mL
Specific Conductance	Plastic/Glass	≤ 6°C	None	28 days	250 mL
Sulfate	Plastic/Glass	≤ 6°C	None	28 days	250 mL
Sulfide	Plastic/Glass	≤ 6°C	Zinc acetate plus NaOH to pH>9	7 days	500 mL
Sulfite	Plastic/Glass	None	None	15 min. <sup>6</sup>	200 mL
Surfactants	Plastic/Glass	≤ 6°C	None	48 hours	1 L
Temperature	Plastic/Glass	None	None	N/A	100 mL
Turbidity	Plastic/Glass	≤ 6°C	None	48 hours	1 L

**Key to Table**

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤ 6°C until compositing and sample splitting is completed.

**Key to Table**

3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. May also be collected in quartz or PTFE Plastic.
6. For compliance testing, the analysis must be performed in the field at the time of analysis. If transported to the laboratory for analysis, the analysis will be performed as soon as practical and reported qualified.
7. Should only be used in the presence of residual chlorine. (Alternatively, sodium arsenite may be used.)
8. H<sub>2</sub>SO<sub>4</sub> to a pH <2 is also acceptable.
9. Except Mercury and Hexavalent Chromium.
10. For dissolved metals, samples must be filtered on site before adding HNO<sub>3</sub> preservative (or before shipping to laboratory).
11. Samples collected for determination of trace level mercury (100 ng/L) using EPA 1631 must be collected in tightly capped Fluor polymer or glad bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipping, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
12. Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) may also be used.
13. Should have glass lid or top.
14. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤ °C" is used in place of the "4 °C" and "<4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).
15. Holding time is 24 hours if pH adjustment is not performed.
16. In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH. If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered (with sulfide treatment by laboratory) and qualify the results in the final report.
17. It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
18. Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.



**Table 22-4**  
**Holding Times, Preservation and Container Requirements: NPDES - Organic**

PARAMETER	CONTAINER	PRESERVATION <sup>1,2</sup>		HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
		Temp. <sup>15</sup>	Chemical		
Purgeable Halocarbons	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	14 days	40 mL
Purgeable Aromatic Hydrocarbons	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , HCl to pH<2	14 days <sup>6</sup>	40 mL
Acrolein and Acrylonitrile	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , adjust pH to 4-5 <sup>7</sup>	14 days	40 mL
Phenols <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days <sup>8</sup>	1 L
Benzidines <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days <sup>8, 11</sup>	1 L
Phthalate esters <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	None	7 days <sup>8</sup>	1 L
Nitrosamines <sup>9,12</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5,13</sup>	7 days <sup>8</sup>	1 L
PCBs <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	None	1 year <sup>8</sup>	1 L
Nitroaromatics and Isophorone <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5,13</sup>	7 days <sup>8</sup>	1 L
Polynuclear Aromatic Hydrocarbons <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5,13</sup>	7 days <sup>8</sup>	1 L
Haloethers <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days <sup>8</sup>	1 L
Chlorinated Hydrocarbons <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	None	7 days <sup>8</sup>	1 L
CDD/CDFs <sup>9</sup> – Aqueous: Field/Lab Preservation	Glass	≤ 6°C	pH <9, 0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	1 year	1 L
CDD/CDFs <sup>9</sup> – Solids/Mixed Phase/ - Field Preservation	Glass	≤ 6°C	None	7 days	1 L
CDD/CDFs <sup>9</sup> – Tissue – Field Preservation	Glass	≤ 6°C	None	24 hours	
CDD/CDFs <sup>9</sup> – Solids/Mixed Phase/Tissue - Lab Preservation	Glass	< -10°C	None	1 year	1 L
Pesticides <sup>9</sup>	Glass	≤ 6°C	pH 5-9 <sup>14</sup>	7 days <sup>8</sup>	1 L



**Key to Table**

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at  $\leq 6^{\circ}\text{C}$  until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid ( $\text{HNO}_3$ ) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. With Teflon lined septum.
5. Should only be used in the presence of residual chlorine. Ascorbic may be used instead.
6. Samples receiving no pH adjustments must be analyzed within 7 days. If 2-chlorovinylethylether is a target analyte, the sample should not be acidified.
7. The pH adjustment is not required if acrolein is not being measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.
8. 7 days until extraction, 40 days after extraction. (PCB only – 1 year after extraction)
9. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more categories, the sample may be preserved by cooling to  $\leq 6^{\circ}\text{C}$  reducing residual chlorine with 0.0008 % sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine) and footnotes 10 and 11(re the analysis of Benzidine).
10. If 1,2-diphenylhydrazine is likely to be present, adjust pH to of the sample to  $4.0 \pm 0.2$  to prevent rearrangement to benzidine.
11. Extracts may be stored up to 30 days before analysis if storage temperature is  $< 0^{\circ}\text{C}$ .
12. For the analysis of diphenylnitrosamine, add 0.008 %  $\text{Na}_2\text{S}_2\text{O}_3$  and adjust pH to 7-10 with NaOH within 24 hours of sampling.
13. Store in dark.
14. The pH adjustment may be performed upon receipt in the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.0008 %  $\text{Na}_2\text{S}_2\text{O}_3$ .
15. Aqueous samples must be preserved at  $\leq 6^{\circ}\text{C}$  unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of " $\leq^{\circ}\text{C}$ " is used in place of the " $4^{\circ}\text{C}$ " and " $<4^{\circ}\text{C}$ " sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to  $6^{\circ}\text{C}$  may not be used to meet the  $\leq 6^{\circ}\text{C}$  requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

**Table 22-5.**  
**Holding Times, Preservation and Container Requirements: NPDES - Radiological**

PARAMETER	CONTAINER	PRESERVATION <sup>1,2</sup>		HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
		Temp.	Chemical		
Alpha, Beta, Radium	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	6 months	1 L

**Key to Table**

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

**Table 22-6.**  
**Holding Times, Preservation and Container Requirements: RCRA - Aqueous**

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION <sup>2,3</sup>		HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
		Temp. <sup>12</sup>	Chemical		
Chloride	Plastic/Glass	4°C	None	28 days	100 mL
Cyanide -Total	Plastic/Glass	4°C	NaOH to pH >12 <sup>5</sup>	14 days	250 mL
Cyanide -Amenable	Plastic/Glass	4°C	NaOH to pH >12 <sup>5</sup>	14 days	250 mL
Hydrogen Ion (pH)	Plastic/Glass	4°C	None	24 hours <sup>11</sup>	100 mL
Nitrate	Plastic/Glass	4°C	None	48 hours	28 days
Oil and Grease	Glass	4°C	HCl	28 days	1 L
Organic carbon (TOC)	Plastic/Glass	4°C	pH to <2 <sup>6</sup> Store in dark	28 days	28 days
Sulfate	Plastic/Glass	4°C	None	28 days	400 mL
Sulfide	Plastic/Glass	4°C	Add Zn Acetate	7 days	400 mL
Chromium VI	Plastic/Glass	4°C	None	24 hours	250 mL
Mercury	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	28 days	250 mL
Other Metals	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>15</sup>	6 months	250 mL
Acrolein and Acrylonitrile	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> Adjust pH to 4-5 <sup>13</sup>	14 days	1 L
Benzidines	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Chlorinated Hydrocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Dioxins and Furans	Glass <sup>10</sup>	4°C	None	30 days <sup>8</sup>	1 L
Haloethers	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Nitroaromatics and cyclic ketones	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Nitrosamines	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Organochlorine Pesticides	Glass <sup>10</sup>	4°C	None	7 days <sup>8</sup>	1 L
Organophosphorus Pesticides	Glass <sup>10</sup>	4°C	Adjust pH <sup>9</sup>	7 days <sup>8</sup>	1 L
PCBs	Glass <sup>10</sup>	4°C	None	None <sup>14</sup>	1 L
Phenols	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION <sup>2,3</sup>		HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
		Temp. <sup>12</sup>	Chemical		
Phthalate Esters	Glass <sup>10</sup>	4°C	None	7 days <sup>8</sup>	1 L
Polynuclear Aromatic Hydrocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Purgeable Hydrocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> Adjust pH <2 <sup>2</sup>	14 days	40 mL
Purgeable Halocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	14 days	40 mL
Total Organic Halides (TOX)	Glass <sup>10</sup>	4°C	Adjust pH to <2 with H <sub>2</sub> SO <sub>4</sub>	28 days	1 L
Radiological Tests (Alpha, Beta, Radium)	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	6 months	250 mL

#### Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. If oxidizing agents are present, add 5 mL 0.1 N NaAsO<sub>2</sub> or 0.06 g of ascorbic acid per L. See Cyanide SOP for additional information about other interferences.
6. Adjust pH to <2 with H<sub>2</sub>SO<sub>4</sub>, HCl, or solid NaHSO<sub>4</sub>. Free Chlorine must be removed prior to adjustment.
7. Free Chlorine must be removed by the appropriate addition of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.
8. 7 days until extraction. 40 days after extraction.
9. Adjust pH to 5-8 using NaOH or H<sub>2</sub>SO<sub>4</sub>.
10. With Teflon lined septum.
11. Holding Time is listed as "As Soon as Possible" in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
12. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to ≤ 6°C is acceptable.
13. Based on guidance from EPA MICE, if samples are received without pH adjustment, the holding time is 7 days.
14. Analysis to be completed within 40 days after extraction.
15. Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.

**Table 22-7.**  
**Holding Times, Preservation and Container Requirements: RCRA – Non-Aqueous**

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION Temp. <sup>7</sup>	Chemical	HOLDING TIME <sup>2</sup>	SAMPLE WEIGHT
Chloride	Glass	4°C	None	28 days	50 g
Cyanide -Total	Glass	4°C	None	14 days	50 g
Cyanide - Amenable	Glass	4°C	None	14 days	50 g
Hydrogen Ion (pH)	Glass	4°C	None	7 days <sup>6</sup>	50 g
Nitrate	Glass	4°C	None	N/A	50 g
Oil and Grease	Glass	4°C	None	28 days	50 g
Sulfide	Glass	4°C	Add Zn Acetate, zero headspace	7 days	50 g
Chromium VI	Glass	4°C	None	30 days	50 g
Mercury	Plastic/Glass	None	None	28 days	50 g
Other Metals	Plastic/Glass	None	None	6 months	50 g
Acrolein and Acrylonitrile	Glass <sup>4</sup>	4°C	None	14 days	50 g
Benzidines	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Chlorinated Hydrocarbons	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Dioxins and Furans	Glass <sup>4</sup>	4°C	None	30 days <sup>3</sup>	50 g
Haloethers	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Nitroaromatics and cyclic ketones	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Nitrosamines	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Organochlorine Pesticides	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Organophosphorus Pesticides	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
PCBs	Glass <sup>4</sup>	4°C	None	None <sup>8</sup>	50 g
Phenols	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Phthalate Esters	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Polynuclear Aromatic Hydrocarbons	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION		HOLDING TIME <sup>2</sup>	SAMPLE WEIGHT
		Temp. <sup>7</sup>	Chemical		
Purgeable Hydrocarbons	Glass <sup>4</sup>	4°C	None	14 days <sup>5</sup>	50 g
Purgeable Halocarbons	Glass <sup>4</sup>	4°C	None	14 days <sup>5</sup>	50 g
Total Organic Halides (TOX)	Glass <sup>4</sup>	4°C	None	28 days	50 g

**Key to Table**

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. 14 days until extraction. 40 days after extraction.
4. With Teflon Lined Septum.
5. See Volatile SOP for more detailed preservation requirements.
6. Holding Time is listed as "As Soon as Possible" in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
7. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to  $\leq 6^{\circ}\text{C}$  is acceptable.
8. Analysis to be completed within 40 days after extraction.

**Table 22-8.**  
**Holding Times, Preservation and Container Requirements: Air Samples**

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION		HOLDING TIME <sup>2</sup>	SAMPLE WEIGHT
		Temp.	Chemical		
Volatile Organics	Summa Canister	None	None	30 days	6L or 1L
Volatile Organics	Tedlar Bag	None	None	72 hrs <sup>3,4</sup>	1 L

**Key to Table**

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. Holding Time is based on SW 846 Method 0040 "SAMPLING OF PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS FROM COMBUSTION SOURCES USING TEDLAR® BAGS". Some states specifically enforce this holding time (e.g. Florida, New Jersey) and others have not specified this information in their regulatory requirements.
4. The holding time is 72 hours unless the laboratory has a documented validation study that indicates a longer HT is acceptable for the analytes of interest.

## **SECTION 23. HANDLING OF SAMPLES**

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

### **23.1 Chain of Custody (COC)**

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

#### **23.1.1 Field Documentation**

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The



sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

### **23.1.2     Legal / Evidentiary Chain-of-Custody**

If samples are identified for legal/evidentiary purposes on the COC, legal COCs will be generated per the Manual for Certification of Laboratories Analyzing Drinking Water, Fifth Edition, January 2005, Appendix A, and SOP No. WS-QA-0003, "Sample Receipt and Procedures".

## **23.2        Sample Receipt**

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections and in SOP No. WS-QA-0003, "Sample Receipt and Procedures".

### **23.2.1     Laboratory Receipt**

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the lot receipt checklist and within the non-conformance program and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record. Laboratory receipt procedures are described in more detail in SOP No. WS-QA-0003.

### 23.2.1.1 Unique Sample Identification

When samples arrive at the laboratory, they are assigned a tracking ID consisting of the Lot ID and a sample number. The lot ID is a 9 character alphanumeric identifier, with the composition as follows:

Character Position	Valid Values	Significance
First	"G"	Assigned to TestAmerica West Sacramento, to distinguish from other laboratories using the Quantims LIMS.
Second	0-9	Last digit of the year in which the samples are received.
Third	A – L	Corresponds to the month in which the samples are received. A = January, B= February, etc.
Fourth & Fifth	01 – 31	Corresponds to the day of the month in which samples are received.
Sixth	0	Separator between day and sequence values.
Seventh through Ninth	400 – 999	Sequence value denoting the order in which lots were logged into Quantims on a given day. Each lot will have a unique sequence number, no matter which Quantims Laboratory logged in the lot.

From the table above, the lot ID G0F100544 is for TestAmerica West Sacramento, logged in in 2010, June, the tenth day, and sequence number 544 for that day.

Once a lot ID is assigned and samples are individually logged in, a workorder(5) and bottle number are assigned to each container. In addition, workorder(8) values are assigned for each analysis requested. The workorder(8) is used to distinguish extracts and digestates produced from the sample. The workorder(5) consists of 5 alphanumeric characters assigned by the LIMS. The LIMS assigns these values sequentially as samples are logged in across the network, using 0-9, and letters of the alphabet excluding "B", "O" and "I", as these may be mistaken for numbers. In addition, sample containers are labeled with the lot ID and a sample number (from 1 to 99).

The workorder(8) consists of the workorder(5), plus 3 additional alphanumeric characters, beginning with "1AA". The first digit of the additional characters denotes the analysis number, such that "1" is the first analysis, "2" is a reanalysis, etc. The second and third characters are assigned sequentially as test requests are added to the sample.

### 23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

**23.3.1** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

**23.3.2** Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.
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Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. WS-QA-0003.

## **23.4      Sample Storage**

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. Empty sample containers are marked as "DIT" (destroyed in testing) on the sample receiving check out form and are disposed by the analytical staff. All samples are kept in the refrigerators for 30 days past invoicing, unless other arrangements have been made with the client.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only.

Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

### **23.5      Hazardous Samples and Foreign Soils**

Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

### **23.6      Sample Shipping**

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

**Note:** If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

### **23.7      Sample Disposal**

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: WS-EHS-001, "Waste Disposal"). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or

deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

### Example: Chain of Custody (COC)

[illegible]

**Figure 23-2. Example: Sample Acceptance Policy**

NELAC and TestAmerica West Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. TestAmerica West Sacramento will review your sample shipment against those requirements as listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

TestAmerica West Sacramento requirements are as follows:


- ✓ Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples, shall be provided.
- ✓ Samples must be accompanied by written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law.
- ✓ Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- ✓ Drinking waters samples for Method 1613B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- ✓ The samples shall arrive at the laboratory with adequate remaining holding time for the analyses requested.
- ✓ Sufficient sample volume must be available to perform the requested analyses.
- ✓ Received samples must not exhibit obvious signs of damage, contamination or inadequate preservation.
- ✓ For samples undergoing chemical warfare degradate analysis, the sample must be screened for agent prior to shipment in accordance with appendix 10 of our Sample Receipt Procedure (WS-QA-0003).
- ✓ Samples containing mammalian tissue will not be accepted without prior coordination with a project manager. Additional conditions for receipt and handling of tissue are outlined in appendix 11 of our Sample Receipt Procedure (WS-QA-0003).

The laboratory will notify the client/Project Manager upon sample receipt if the samples fail to meet any of the above requirements.

When completing the chain of custody form, please do not forget to sign your name in the "relinquished by" box.



Figure 23-3. Example: Cooler Receipt Form



THE LEADER IN ENVIRONMENTAL TESTING

**LOT RECEIPT CHECKLIST**  
TestAmerica West Sacramento

CLIENT \_\_\_\_\_ PM \_\_\_\_\_ LOG # \_\_\_\_\_

LOT# (QUANTIMS ID) \_\_\_\_\_ QUOTE# \_\_\_\_\_ LOCATION \_\_\_\_\_

DATE RECEIVED \_\_\_\_\_ TIME RECEIVED \_\_\_\_\_

DELIVERED BY: ☐ FEDEX ☐ ON TRAC ☐ CLIENT

☐ GOLDENSTATE ☐ UPS ☐ GO-GETTERS ☐ OTHER

☐ TAL COURIER ☐ TAL SF ☐ VALLEY LOGISTICS

CUSTODY SEAL STATUS ☐ INTACT ☐ BROKEN ☐ N/A

CUSTODY SEAL #(S) \_\_\_\_\_

SHIPPING CONTAINER(S) ☐ TAL ☐ CLIENT ☐ N/A

COC #(S) \_\_\_\_\_

TEMPERATURE BLANK Observed: \_\_\_\_\_ Corrected: \_\_\_\_\_

SAMPLE TEMPERATURE - (TEMPERATURES ARE IN °C)

Observed: \_\_\_\_\_ Average \_\_\_\_\_ Corrected Average \_\_\_\_\_

LABORATORY THERMOMETER ID: \_\_\_\_\_

IR UNIT: #4 ☐ #5 ☐ OTHER \_\_\_\_\_

Checked (✓)

	Initials	Date
pH MEASURED <input type="checkbox"/> YES <input type="checkbox"/> ANOMALY <input type="checkbox"/> N/A		<input type="checkbox"/>
LABELED BY _____		<input type="checkbox"/>
LABELS CHECKED BY _____		<input type="checkbox"/>
PEER REVIEW _____ <input type="checkbox"/> NA		
SHORT HOLD TEST NOTIFICATION		<input type="checkbox"/>
SAMPLE RECEIVING		<input type="checkbox"/>
WETCHEM <input type="checkbox"/> N/A		<input type="checkbox"/>
VOA-ENCORES <input type="checkbox"/> N/A		<input type="checkbox"/>
<input type="checkbox"/> METALS NOTIFIED OF FILTER/PRESERVE VIA VERBAL & EMAIL <input type="checkbox"/> N/A		<input type="checkbox"/>
<input type="checkbox"/> COMPLETE SHIPMENT RECEIVED IN GOOD CONDITION WITH <input type="checkbox"/> N/A APPROPRIATE TEMPERATURES, CONTAINERS, PRESERVATIVES		<input type="checkbox"/>
<input type="checkbox"/> CLOUSEA <input type="checkbox"/> TEMPERATURE EXCEEDED (2 °C – 6 °C) <sup>1</sup> <input type="checkbox"/> N/A		
<input type="checkbox"/> WET ICE <input type="checkbox"/> BLUE ICE <input type="checkbox"/> GEL PACK <input type="checkbox"/> NO COOLING AGENTS USED <input type="checkbox"/> PM NOTIFIED		

Initials \_\_\_\_\_

Date \_\_\_\_\_

Notes \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\*1 Acceptable temperature range for State of Wisconsin samples is ≤4°C.





## Bottle Lot Inventory

Lot ID: \_\_\_\_\_

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
VOA <sup>h</sup>																				
VOA <sup>h</sup> n <sup>h</sup>																				
AGB																				
AGBs																				
250AGB																				
250AGBs																				
250AGBn																				
500AGB																				
____AGJ																				
500AGJ																				
250AGJ																				
125AGJ																				
____CGJ																				
500CGJ																				
250CGJ																				
125CGJ																				
PJ																				
PJn																				
500PJ																				
500PJn																				
500PJna																				
500PJzn/na																				
250PJ																				
250PJn																				
250PJna																				
250PJzn/na																				
Acetate Tube																				
____CT																				
Enbore																				
Folder/filter																				
PUF																				
Petri/Filter																				
XAD Trap																				
Ziploc																				

h = hydrochloric acid    s = sulfuric acid    na = sodium hydroxide    n = nitric acid    zn = zinc acetate

Number of VOAs with air bubbles present / total number of VOA's

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## SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

### 24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

### 24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

### 24.3 Negative Controls

**Table 24-1. Example – Negative Controls**

Control Type	Details
Method Blank (MB)	<p>are used to assess preparation and analysis for possible contamination during the preparation and processing steps.</p> <p>The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.</p> <p>The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.</p> <p>The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).</p> <p>Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.</p>
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

**Table 24-1. Example – Negative Controls**

Control Type	Details
Trip Blank <sup>1</sup>	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks <sup>1</sup>	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks <sup>1</sup>	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

<sup>1</sup> When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

## 24.4 **Positive Controls**

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

### 24.4.1 **Method Performance Control - Laboratory Control Sample (LCS)**

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- 
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

## 24.5 Sample Matrix Controls

**Table 24-3. Sample Matrix Control**

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency <sup>1</sup>	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency <sup>1</sup>	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates <sup>2</sup>	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency <sup>1</sup>	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency <sup>1</sup>	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

<sup>1</sup> See the specific analytical SOP for type and frequency of sample matrix control samples.

<sup>2</sup> LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

## 24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.



Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking  $\pm 3$  Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- The maximum acceptable recovery limit will be 150%. Some specific methods or SOPs may allow for higher recoveries.
- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- If either the high or low end of the control limit changes by  $\leq 5\%$  from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**24.6.1** The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. See Policy WS-PQA-003 for further details.

**24.6.2** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.

- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for TNI and Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).
- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

**24.6.3** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

**24.6.4** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

## **24.7 Additional Procedures to Assure Quality Control**

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.



## **SECTION 25. REPORTING RESULTS**

### **25.1 Overview**

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

### **25.2 Test Reports**

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed or prepared electronically on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

**25.2.1** A report title (e.g. Analytical Report For Samples) with a "sample results" column header.

**25.2.2** Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

**25.2.3** A unique identification of the report (e.g. work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as page # of ##, where the first number is the page number and the second is the total number of pages.

**25.2.4** A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.
- In most cases, the applicable COC is an integral part of the report.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

- 25.2.5** The name and address of client and a project name/number, if applicable.
- 25.2.6** Client project manager or other contact
- 25.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.
- 25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.
- 25.2.9** Date reported or date of revision, if applicable.
- 25.2.10** Method of analysis including method code (EPA, Standard Methods, etc).
- 25.2.11** Reporting limit.
- 25.2.12** Method detection limits (if requested)
- 25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).
- 25.2.14** Sample results.
- 25.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.
- 25.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).
- 25.2.17** A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.
- 25.2.18** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.
- 25.2.19** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.
- 25.2.20** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.
- 25.2.21** When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.
- 25.2.22** The laboratory includes a cover letter.

**25.2.23** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**25.2.24** When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

**25.2.25** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**25.2.26** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report). A complete report must be sent once all of the work has been completed.

**25.2.27** Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

**25.2.28** A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

### **25.3      Reporting Level or Report Type**

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level II is a report with the features described in Section 25.2 above, plus summary information, including results for the method blank reported to the laboratory MDL if required, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. No raw data is provided unless it is necessary to provide the relevant calibration information.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in electronic deliverable form via e-mail, posting to an FTP site, or CD ROM. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

### **25.3.1 Electronic Data Deliverables (EDDs)**

EDDs are routinely offered as part of TestAmerica's services. TestAmerica West Sacramento offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD, and a copy filed on the QA share of the local server.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

### **25.4 Supplemental Information for Test**

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

**Note:** Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This

necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of “interpretation” of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

## **25.5      Environmental Testing Obtained From Subcontractors**

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory’s original report stationery and the report includes any accompanying documentation.

## **25.6      Client Confidentiality**

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity’s proprietary rights will not be released.

**Note:** This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

**Note:** Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

**25.6.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

*This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-916-373-5600*

*(or for e-mails: please notify us immediately by e-mail or by phone (1-916-373-5600) and delete this material from any computer).*

## **25.7      Format of Reports**

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## **25.8      Amendments to Test Reports**

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "Amend". The revised report will have the word "revised" or "amended" next to the date in the footer.

When the report is re-issued, a notation of "Amended" is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example: Report was revised on 11/3/11 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/11.*

## **25.9      Policies on Client Requests for Amendments**

### **25.9.1      Policy on Data Omissions or Reporting Limit Increases**

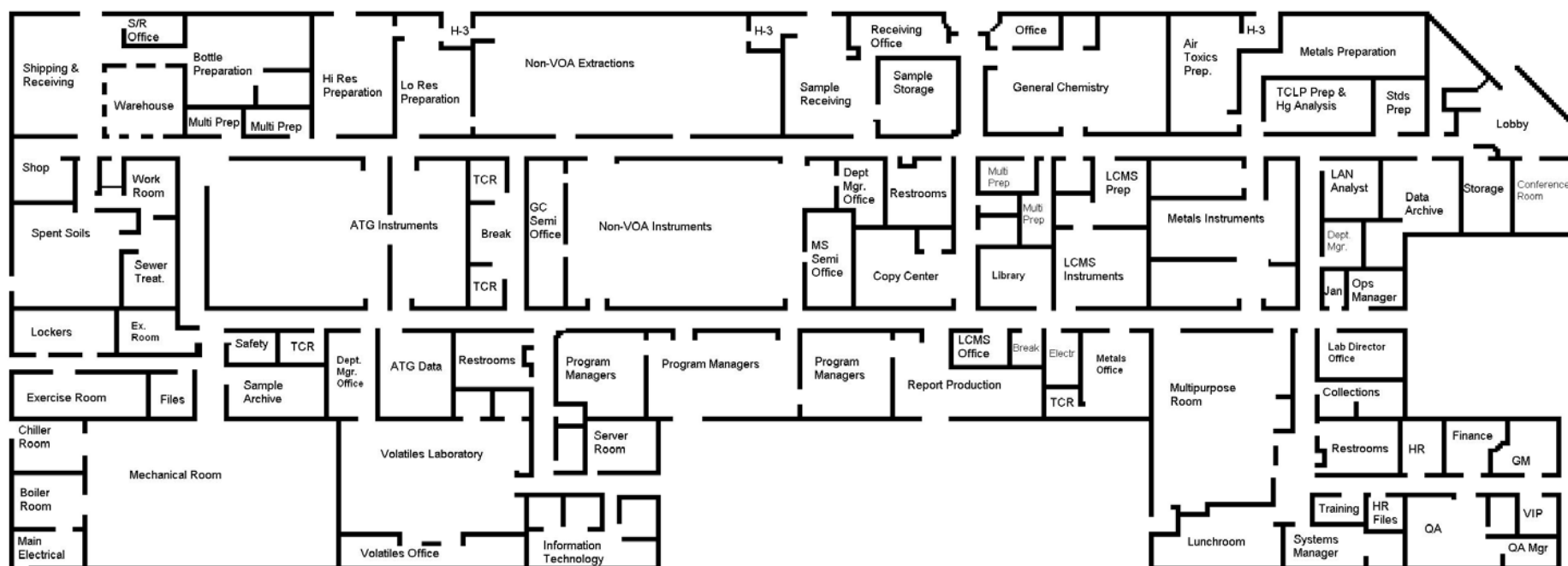
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

### **25.9.2      Multiple Reports**

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

## Appendix 1. Laboratory Floor Plan



<u>Facility Size</u>	<u>Square Feet</u>
Total Area	66,000
Lab Area	43,000
Storage Area	5,200
	<u>Linear Feet</u>
Bench Top	3,000
Hoods	500



## Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

### Glossary:

**Acceptance Criteria:** Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

**Accreditation:** The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

**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. (QAMS)

**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.

**Analytical Uncertainty:** A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

**Assessment:** The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

**Audit:** A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

**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 (1) to twenty (20) environmental samples of the same quality systems 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 twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

**Bias:** The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample’s true value). (TNI)

**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. (ASQC)

**Calibration:** A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)



- 1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).
- 2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

**Calibration Curve:** The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

**Calibration Standard:** A substance or reference material used to calibrate an instrument (QAMS)

**Certified Reference Material (CRM):** A reference material accompanied by certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

**Chain of Custody (COC) Form:** Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

**Compromised Samples:** Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

**Confidential Business Information (CBI):** Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

**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. (TNI)

**Conformance:** An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

**Correction:** Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

**Corrective Action:** The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

**Data Audit:** A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

**Data Reduction:** The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

**Deficiency:** An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

**Demonstration of Capability:** A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

**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. (ASQC)

**Duplicate Analyses:** The analyses or measurements of the variable of interest performed identically on two subsamples 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. (EPA-QAD)

**Equipment Blank:** Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

**External Standard Calibration:** Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

**Field Blank:** Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

**Field of Accreditation:** Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

**Holding Times:** The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

**Internal Standard:** A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

**Internal Standard Calibration:** Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

**Instrument Blank:** A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

**Instrument Detection Limit (IDL):** The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is  $\pm 100\%$ . The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

**Laboratory Control Sample (however named, 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 or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. 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.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

**Least Squares Regression (1<sup>st</sup> Order Curve):** The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

**Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]:** A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

**LOD Verification [a.k.a., MDL Verification]:** A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

**Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]:** The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

**(QS) 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 or Saline/Estuarine. Includes surface water, groundwater, effluents, and TCLP or other extracts.

*Drinking Water:* Any aqueous sample that has been designated as 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.

*Biological Tissue:* Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

*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 & Emissions:* 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 sorbant tube, impinger solution, filter, or other device. (TNI)

**Matrix Spike (spiked sample or fortified sample):** A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test

result 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 replicate matrix spike prepared 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 and 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. (40 CFR Part 136, Appendix B)

**Negative Control:** Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

**Non-conformance:** An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

**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.

**Positive Control:** Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

**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. (TNI)

**Preservation:** Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

**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. (TNI)

**Proficiency Testing Program:** The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

**Proficiency Test Sample (PT):** A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

**Quality Assurance:** An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

**Quality Assurance [Project] Plan (QAPP):** A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

**Quality Control:** The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

**Quality Control Sample:** A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

**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. (TNI)

**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 activities. (TNI)

**Raw Data:** The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

**Record Retention:** The systematic collection, indexing and storing of documented information under secure conditions.

**Reference Material:** Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

**Reference Standard:** Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

**Sampling:** Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

**Second Order Polynomial Curve (Quadratic):** The 2<sup>nd</sup> order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2<sup>nd</sup> order regression will generate a coefficient of determination (COD or  $r^2$ ) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes,  $r^2$  must be greater than or equal to 0.99.

**Selectivity:** The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

**Sensitivity:** The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

**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 standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

**Standard Operating Procedures (SOPs):** A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

**Storage Blank:** A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

**Surrogate:** A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

**Systems Audit (also Technical Systems Audit):** A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

**Technical Manager:** A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

**Technology:** A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

**Traceability:** The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

**Trip Blank:** A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

**Uncertainty:** A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.



## Acronyms:

A2LA – American Association for Laboratory Accreditation  
ANSI – American National Standards Institute  
ASQ – American Society for Quality  
CAR – Corrective Action Report  
CCB – Continuing Calibration Blank  
CCV – Continuing Calibration Verification  
CF – Calibration Factor  
CFR – Code of Federal Regulations  
COC – Chain of Custody  
DOC – Demonstration of Capability  
DQO – Data Quality Objectives  
DUP - Duplicate  
EHS – Environment, Health and Safety  
EPA – Environmental Protection Agency  
GC - Gas Chromatography  
GC/MS - Gas Chromatography/Mass Spectrometry  
HPLC - High Performance Liquid Chromatography  
ICB – Initial Calibration Blank  
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy  
ICP/MS – ICP/Mass Spectrometry  
ICV – Initial Calibration Verification  
IDL – Instrument Detection Limit  
IH – Industrial Hygiene  
IS – Internal Standard  
LCS – Laboratory Control Sample  
LCSD – Laboratory Control Sample Duplicate  
LIMS – Laboratory Information Management System  
LOD – Limit of Detection  
LOQ – Limit of Quantitation  
MDL – Method Detection Limit  
MDLCK – MDL Check Standard  
MDLV – MDL Verification Check Standard  
MRL – Method Reporting Limit Check Standard  
MS – Matrix Spike  
MSD – Matrix Spike Duplicate  
MSDS - Material Safety Data Sheet  
NELAP - National Environmental Laboratory Accreditation Program  
PT – Performance Testing  
TNI – The NELAC Institute  
QAM – Quality Assurance Manual  
QA/QC – Quality Assurance / Quality Control  
QAPP – Quality Assurance Project Plan  
RF – Response Factor  
RPD – Relative Percent Difference  
RSD – Relative Standard Deviation  
SD – Standard Deviation  
SOP – Standard Operating Procedure  
TAT – Turn-Around-Time  
VOA – Volatiles  
VOC – Volatile Organic Compound

### Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica West Sacramento maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:



#### TestAmerica Certifications

Laboratory	Program	Authority	Identification	Expiration Date
TestAmerica West Sacramento	Alaska UST	Alaska	UST-055	12/08/2011
TestAmerica West Sacramento	DoD ELAP	AZLA	2828-D1	01/31/2012
TestAmerica West Sacramento	NELAC	California	1116CA	01/31/2012
TestAmerica West Sacramento	NELAC	Florida	E87570	08/30/2011
TestAmerica West Sacramento	NELAC	Illinois	200050	03/17/2012
TestAmerica West Sacramento	NELAC	Kansas	E-10375	10/31/2011
TestAmerica West Sacramento	NELAC	Louisiana	30612	08/30/2011
TestAmerica West Sacramento	NELAC	New Jersey	CA005	08/30/2012
TestAmerica West Sacramento	NELAC	New York	11886	04/01/2012
TestAmerica West Sacramento	NELAC	Oregon	CA200005	03/28/2012
TestAmerica West Sacramento	NELAC	Pennsylvania	68-01272	03/31/2012
TestAmerica West Sacramento	NELAC	Texas	T104704399-05-TX	05/31/2012
TestAmerica West Sacramento	NELAC	Utah	QUAN1	01/31/2012
TestAmerica West Sacramento	State Program	Arizona	AZ0708	08/11/2012
TestAmerica West Sacramento	State Program	Arkansas	88-0591	08/17/2011
TestAmerica West Sacramento	State Program	Colorado	N/A	08/31/2011
TestAmerica West Sacramento	State Program	Connecticut	PH-0691	08/30/2011
TestAmerica West Sacramento	State Program	Georgia	980	01/31/2010
TestAmerica West Sacramento	State Program	Guam	N/A	08/31/2011
TestAmerica West Sacramento	State Program	Hawaii	N/A	01/31/2012
TestAmerica West Sacramento	State Program	Michigan	9047	01/31/2012
TestAmerica West Sacramento	State Program	Nevada	CA44	07/31/2011
TestAmerica West Sacramento	State Program	New Mexico	N/A	08/30/2011
TestAmerica West Sacramento	State Program	South Carolina	87014	01/31/2012
TestAmerica West Sacramento	State Program	Virginia	178	08/30/2011
TestAmerica West Sacramento	State Program	Washington	C581	05/05/2012
TestAmerica West Sacramento	State Program	Wisconsin	988204680	08/31/2011
TestAmerica West Sacramento	State Program	Wyoming	8TMS-Q	01/31/2012
TestAmerica West Sacramento	US Fish & Wildlife	US Fish & Wildlife	LE148388-0	02/29/2012
TestAmerica West Sacramento	USDA	USDA	P335-09-00055	03/19/2012
TestAmerica West Sacramento	USEPA UCMR	USEPA UCMR	CA00044	10/18/2011
TestAmerica West Sacramento	West Virginia DEP	West Virginia	334	07/31/2012
TestAmerica West Sacramento	West Virginia DHR (DW)	West Virginia	9930C	12/31/2011

The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.



#### Appendix 4: Listing of Methods Performed

<b>Preparation Only Methods</b>					
<b>Method</b>	<b>Aqueous</b>	<b>Solid</b>	<b>Waste</b>	<b>Biological</b>	<b>Air</b>
<b>Organics</b>					
Calif. CAM-WET	X	X	X		
EPA 1311	X	X	X		
EPA 3510C	X				
EPA 3520C	X				
EPA 3535	X				
EPA 3540B		X			
EPA 3542					X
EPA 3550B		X		X	
EPA 3580A			X		
EPA 3600C	X	X	X		
EPA 3620B	X	X	X		
EPA 3630C	X	X	X		
EPA 3640A	X	X		X	
EPA 5030B	X	X	X		
EPA 5035	X	X	X		
<b>Inorganics</b>					
Calif. CAM WET	X	X	X		
EPA 1311	X	X	X		
EPA 1312 (W)	X	X	X		
EPA 3005A	X				
EPA 3010A	X				
EPA 3050B		X	X	X	

### Organics Methods Performed

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
<b>Volatile Organics</b>	SW846 8260B	X	X	X		
<b>Base Neutrals and Acids (BNAs)</b>	SW846 8270B	X	X	X	X	
	TO-13A					X
	IP-7					X
	EPA 23					X
<b>Organochlorine Pesticides</b>	SW846 8081A	X	X	X	X	
	TO-4A					X
	TO-10A					X
	IP-8					X
	WS-ID-0014	X	X	X	X	
	EPA 1669 (Mod)	X	X	X		
<b>PCBs</b>	EPA 8082	X	X	X	X	
	TO-4A					X
	TO-10A					X
<b>Petroleum Hydrocarbons</b>	EPA 8015B	X	X	X		
	CA LUFT	X	X	X		
	AK101	X	X	X		
	AK102	X	X	X		
	AK103	X	X	X		
	NWTPH-Gx	X	X	X		
	NWTPH-Dx	X	X	X		
	GRO/DRO	X	X	X		
<b>Nitroaromatics and Nitroamines</b>	EPA 8330	X	X	X		X
	EPA 8330A	X	X	X		
	EPA 8330B	X	X	X		
	EPA 8321A (modified)	X	X	X		
	WS-LC-0001	X	X	X		
	WS-LC-0009	X	X	X		
	WS-LC-0010	X	X	X		
<b>Nitrosamines</b>	WS-MS-0012	X	X			
<b>PAHs</b>	EPA 8270C (SIM Isotope dilution)	X	X	X	X	X
	EPA 8270C (SIM)	X	X	X		
	CARB 429	X	X	X	X	X
	TO-13A					X
	IP-7					X
<b>1,4-Dioxane</b>	WS-MS-0010	X				
<b>Alkyl Phenols</b>	WS-MS-0013	X	X		X	
<b>CBSA</b>	WS-LC-0013	X	X			

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
Chemical Warfare Degradates	WS-LC-0004	X	X			
Organosulfur Degradates	EPA 8270C	X	X			
	WS-MS-0003	X	X			
PFOA/PFOS	WS-LC-0020	X	X			
PPCPs (Pharmaceuticals & Personal Care Products)	EPA 1694	X				
Steroids & Hormones	EPA 1698	X				
PCB Congeners	EPA 1668A	X	X	X	X	X
	EPA 1668C	X	X	X	X	X
	CBC1.2	X	X	X		
Dioxins & Furans	EPA 1613B	X	X			
	EPA 8290	X	X	X	X	
	EPA 8290A	X	X	X	X	
	EPA 8280A	X	X	X	X	
	EPA 8280B	X	X	X	X	
	DLFM2.2	X	X	X		
	EPA 0023A					X
	EPA 23					X
	TO-9					X

**Metals Methods Performed**

Parameter	Methods	Aqueous	Solid	Waste	Biological	Air
<b>Trace Metals</b>	EPA 200.7	X				
	EPA 200.8	X				
	EPA 6010B	X	X	X	X	X
	EPA 6020	X	X	X	X	X
	EPA 0060					X
	EPA 12					X
	CARB 12					X
	EPA 29					X
	CARB 436					X
<b>Hardness</b>	SM 2340B	X				
	EPA 200.7	X				
	EPA 200.8	X				
<b>Mercury</b>	EPA 245.1	X				
	EPA 200.8	X				
	EPA 7470A	X				
	EPA 7471A		X	X	X	X
	EPA 101A					X
	ASTM D6784-02					X
	Ontario-Hydro					X
	EPA 0060					X
	EPA 29					X
	CARB 436					X

**Inorganics Methods Performed**

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
<b>Alkalinity (Carbonate, Bicarbonate, Total)</b>	SM 2320B	X				
<b>Ammonia</b>	EPA 350.1	X				X
<b>Bromide</b>	EPA 300.0	X				
	EPA 9056	X	X			
	EPA 9057					X
	EPA 26A					X
	CARB 421					X
<b>Carbon, Total Inorganic</b>	EPA 9060	X	X			
<b>Carbon, Total Organic</b>	EPA 9060	X	X			
	SM 5310 C	X				
<b>Chloride</b>	EPA 300.0	X				
	EPA 9056	X	X			
	EPA 9057					X
	EPA 26A					X
	CARB 421					X
<b>Chromium, Hexavalent</b>	EPA 7196A	X	X			
	EPA 0061					X
	EPA 306					X
	CARB 426					X
<b>Conductivity</b>	EPA 9050A	X				
	SM 2510 B	X				
<b>Cyanide, Free</b>	EPA 9012A	X	X			
	SM 4500 CN E	X				
<b>Cyanide, Total</b>	EPA 335.4	X				
	EPA 9012A	X	X			
	CARB 426					X
<b>Demand, Chemical Oxygen</b>	EPA 410.4	X				
<b>Flouride</b>	EPA 300.0	X	X			
	EPA 9056	X	X			
	SM 4500 F C	X				
	EPA 9057					X
	EPA 26A					X
	CARB 421					X
<b>n-Hexane Extractable Materials</b>	EPA 1664A	X				
	EPA 9070A	X				
	EPA 9071B		X			
<b>Moisture</b>	ASTM 2216		X			

<b>Nitrate</b>	EPA 353.2	X				
	EPA 300.0	X				
	EPA 9056	X	X			
	CARB 421					X
<b>Nitrate-Nitrite</b>	EPA 353.2	X				
<b>Nitrite</b>	EPA 353.2	X				
	EPA 300.0	X				
	EPA 9056	X	X			
	CARB 421					X
<b>Nitrocellulose</b>	EPA 353.2	X	X			
	WS-WC-0050	X	X			
<b>Total Kjeldahl Nitrogen</b>	EPA 351.2	X				
<b>Orthophosphate</b>	EPA 300.0	X				
	EPA 9056	X	X			
<b>Particulates in Air</b>	EPA 5					X
	40 CFR Part 50					X
<b>Perchlorate</b>	EPA 314.0	X				
	EPA 331.0	X				
	EPA 6850	X	X			
	WS-LC-0012	X	X			
<b>pH</b>	SM 4500 H+ B	X				
	EPA 150.2	X				
	EPA 9040A	X				
	EPA 9041A	X				
	EPA 9045C		X	X		
<b>Phosphorus, Total</b>	EPA 365.4	X				
<b>Solids, Total</b>	SM 2540 B	X				
<b>Solids, Total Dissolved</b>	SM 2540 C	X				
<b>Solids, Total Suspended</b>	SM 2540 D	X				
<b>Sulfate</b>	EPA 300.0	X				
	EPA 9065	X				
<b>Sulfide</b>	SM 4500 S2- D	X				

## Appendix 5 . Data Qualifiers

Qualifier Organic	Qualifier Inorganic	Footnote
U	U	Analyte analyzed for but was not detected.
G	G	Elevated reporting limit. The reporting limit is elevated due to matrix interference.
J	B	Estimated result. Result is less than RL.
E	I	Estimated result. Result concentration exceeds the calibration range.
B	J	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
P	*	Relative percent difference (RPD) is outside stated control limits.
a	N	Spiked analyte recovery is outside stated control limits.
*		Surrogate recovery is outside stated control limits.
PG		The percent difference between the original and confirmation analyses is greater than 40%.

## **Attachment B: Health and Safety Plan**



Prepared for:

U.S. Department of the Interior  
National Park Service  
401 West Hillcrest Drive  
Thousand Oaks, CA 91360

## Health and Safety Plan for Soil Characterization

Devils Postpile National Monument  
Madera County, California

June 17, 2013

Prepared By:



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Date



*Holly Trejo*  
\_\_\_\_\_  
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Project Manager

June 17, 2013  
\_\_\_\_\_  
Date

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## ACRONYMS AND ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
CA	California
CCHLS	California Human Health Screening Levels
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CNS	Central Nervous System
CFR	Code of Federal Regulations
CPR	Cardiopulmonary resuscitation
DEPO	Devils Postpile National Monument
EE/CA	Engineering Evaluation and Cost Analysis
ECM	Environmental Cost Management, Inc.
HASP	<i>Health and Safety Plan</i>
HRS	Hazard Ranking System
IDLH	Immediately dangerous to life or health
JHA	Job hazard analysis
mg/kg	Milligram per kilogram
MSDS	Material Safety Data Sheets
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NIOSH	National Institute for Occupational Safety and Health
NPS	National Park Service
PA	Preliminary Assessment
PEL	Permissible exposure limit
PM	Project Manager
PPE	Personal protective equipment
REL	Recommended exposure limit
SEIR	Supervisor's Employee Injury Report
SSHO	Site Safety and Health Officer
TLV	Threshold limit value
TTLC	Total Threshold Limit Concentration
TWA	Time-weighted average (8-hour)
USEPA	United States Environmental Protection Agency

## 1. INTRODUCTION

Environmental Cost Management, Inc. (ECM) prepared this *Health and Safety Plan* (HASP) for the National Park Service (NPS) Devils Postpile National Monument (DEPO), 100,000 gallon steel water tank site(**Figure 1**).

ECM prepared this HASP to ensure the safety and well-being of all field personnel conducting work in accordance with site-specific project goals and objectives. All personnel completing field activities must review and sign this HASP to confirm understanding of the project safety goals.

This HASP presents known or anticipated environmental, health, and safety concerns at DEPO. Some general safety concerns include:

- Physical hazards;
- Meteorological hazards such as flash flood or lightning;
- Physiological hazards such as heat stress or dehydration;
- Biological hazards such as bee stings or snake bites; and
- Chemical hazards.

NPS is the lead regulatory agency. NPS is conducting a Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) non-time critical removal action for the Site. NPS and ECM's subcontractors will operate under their own separately prepared HASPs.

This HASP may act as a stand-alone document for the purpose of addressing Site health and safety concerns. However, this HASP has also been prepared to act as a supplement to any Site-specific investigation plan, such as the Engineering Evaluation & Cost Analysis (EE/CA) Work Plan. Specific details of any proposed field investigation activities (such as laboratory methodologies and field procedures) are presented in an applicable work plan.

**Appendix A** of this HASP presents Material Safety Data Sheets (MSDS) for hazardous materials which may be found in or around the Site during investigation activities. MSDS may be amended as new information is obtained during Site investigation activities.

## 2. ORGANIZATIONAL STRUCTURE

The specific responsibilities and authority of management of personnel, as well as safety and health, are detailed in the following paragraphs:

**Program Manager:** Andrew Campbell is the Program Manager (PM) for this project. The PM has responsibility and authority to direct all work operations. The PM coordinates safety and health functions with the Site Safety and Health Officer (SSHO), has the authority to oversee and monitor the performance of the SSHO, and bears ultimate responsibility for the proper implementation of this HASP. The specific duties of the PM are:

- Coordinating the preparation and enactment of a site-specific Work Plan.
- Providing Site supervisor(s) with work assignments and overseeing their performance.
- Coordinating safety and health efforts with the SSHO, and serving as primary Site liaison with public agencies, officials, and contractors.

The qualified alternate Program Manager (PM) for this Site is Wallid Kazi.



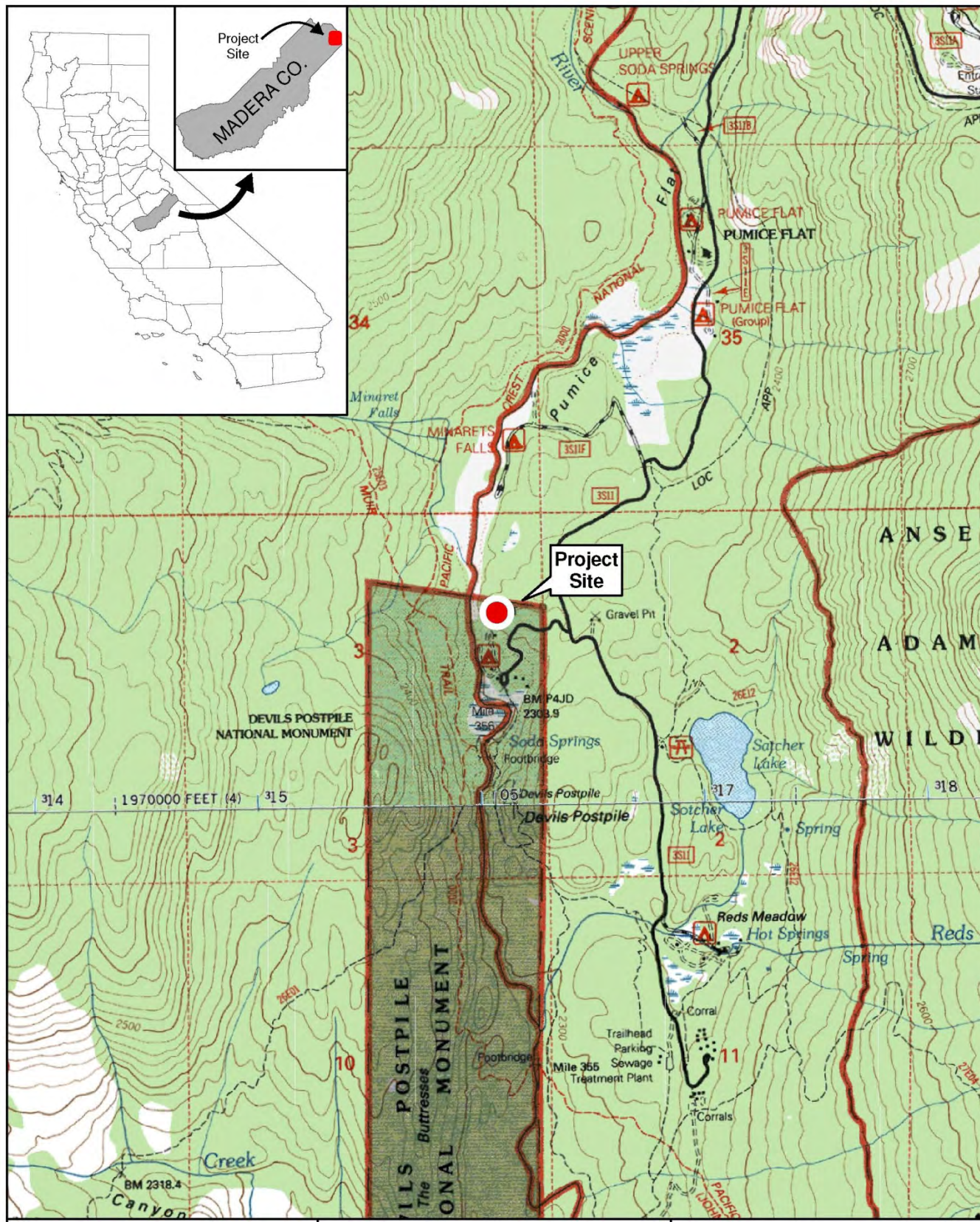


Figure 1: Site Location Map

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**Site Safety and Health Officer:** Holly Trejo is the SSHO for this Site. The SSHO has full responsibility and authority to develop and implement this HASP and to verify compliance. The SSHO reports to the Project Manager. The SSHO is readily accessible during all work operations and has the authority to halt work if unsafe conditions are detected. The specific responsibilities of the SSHO are:

- Managing the safety and health functions on the Site.
- Serving as the Site's point of contact (POC) for safety and health matters.
- Ensuring monitoring, worker training, and effective selection and use of PPE.
- Assessing conditions for unsafe acts and providing corrective action.
- Assisting the preparation and review of this HASP.
- Maintaining effective safety and health records as described in this HASP.
- Coordinating with Site Supervisor(s), DEPO Park Headquarters, and others as necessary for safety and health efforts.

The qualified alternate SSHO for this Site is Andrew Campbell.

**Site Supervisor:** Holly Trejo is the Site Supervisor. The Site Supervisor is responsible for field operations and reports to the SSHO. The Site Supervisor ensures the implementation of the HASP requirements and procedures in the field. The specific responsibilities of the Site Supervisor are:

- Executing the work plan and schedule as detailed by the PM.
- Coordination with the SSHO on safety and health.
- Ensuring work is in compliance with the requirements of this HASP.

The qualified alternate Site Supervisor for this Site is Andrew Campbell.

**Table 1: Contact Telephone Numbers**

Contact	Title	Phone number
Police Emergency		911
Fire Emergency		911
Jonathan Winters	DEPO Contact Safety & Occupational Health Manager	Office: (760) 934-8170 Cell: (317) 518-3190
Wallid Kazi	ECM President	Office: (714) 662-2757 Cell: (404) 886-3854
Andrew Campbell	ECM PM	Office: (916) 241-9290 Cell: (916) 826-3659
Holly Trejo	ECM SSHO	Office: (510) 964-4399 Cell: (510) 685-6268
Chris McCormack	ECM Site Supervisor	Cell: (925) 584-2416



### **3. SITE DESCRIPTION AND BACKGROUND**

DEPO is located along the Middle Fork of the San Joaquin River Valley in the south eastern Sierra Nevada, approximately 2 miles southwest of Mammoth Mountain ski resort in Madera County, California at 37.629 N Longitude and 119.0847 W Latitude.

Located on the western slope of the Sierra Nevada range between 7,200 and 8,200 feet, DEPO contains an interesting assemblage of flora, fauna and geology, for which the monument was set aside. DEPO's landscape is a result of eruptions and uniform cooling of basalt lava that created an impressive wall of columns. Later, a glacial event exposed the columns and polished smooth the top of this formation, enhancing the pattern of hexagons that resulted from the mineral composition of the lava.

In 2005, DEPO contracted with AA-1 Services, Inc. of Paramount, California to sandblast and recoat the exterior of the 100,000 gallon potable water tank. The tank site is located on a slope north east of the campground (Figure 2). Reportedly the tank had not been repainted since its installation prior to 1940 (Fernandes, 2008). The tank's paint had weathered to the point that paint was peeling and flaking (Photograph 2). AA-1 Service constructed what appeared to be negative pressure containment system by wrapping scaffolding surrounding the tank with a plastic material. The primary type of waste generated on site was a onetime release of lead based paint chips related to the sandblasting operations for external tank cleaning in preparation recoating. It is also likely that some amount of the blasting materials was also released to the soil during the blasting operations in 2005. However the sandblasting material is not considered an environmental hazard.

#### **3.1 ENVIRONMENTAL BACKGROUND**

The following sections describe the past and future activities relating to the Site, including field investigations, sampling, and assessment.

##### **3.1.1 Past Activities**

In 2008, at the request of the NPS, Provost and Pritchard Consulting Group (P&P) conducted a Preliminary Assessment<sup>1</sup> (PA) in general accordance with the CERCLA guidance manual for the 2005 release of lead based paint chips and sand blasting debris, at the 100,000 gallon above-ground potable water tank at DEPO. The objective of the PA was to identify past and present practices related to the historic release and evaluate the site's Hazard Ranking System score (HRS).

The scope of the investigation included review of available records, a site reconnaissance and interviews with DEPO personnel. The investigation focused on the 2005 water tank sandblasting operations activities intended to remove the lead-based paint from the exterior of the tank. The tank was reportedly installed prior to 1940 and had not been repainted since its installation.

In November of 2005, following sandblasting and painting operations, Mr. John Fernandes<sup>1</sup>, DEPO Maintenance Supervisor, collected ten soil samples within the sandblasting containment area to verify the painting contractor's cleanup. The analytical results for samples collected by Mr. Fernandes indicated a maximum lead concentration of 2,100 milligrams per kilogram (mg/kg) and a minimum concentration of 20 mg/kg. Concentrations of lead in site soils are below the California Human Health Screening Levels (CCHLS) of 3,500 mg/kg for commercial/industrial use. However, the average lead concentrations slightly exceed the Total Threshold Limit Concentration (TTLC) of 1,000 mg/kg, as defined in Title 22, California Code of Regulations.

##### **3.1.2 Future Activities**

Additional site characterization is proposed in the EE/CA Work Plan to supplement the understanding of the nature and extent of contamination near the potable water tank at DEPO.

## **4. SITE HAZARDS AND TASK/OPERATION SAFETY**

The following sections discuss the potential physical, chemical, and biological hazards associated with the proposed field activities. There are some general hazards involved with the performance of the field tasks due to the Site conditions and remoteness that include:

- Physical hazards (e.g. slips/trips/falls);
- Physiological hazards such as altitude sickness, heat stress or dehydration;
- Meteorological hazards such as lightning;
- Biological hazards such as bee stings, snake bites, or bears; and
- Chemical hazards.

### **4.1 PHYSICAL HAZARDS**

Physical hazards present the most significant health hazards, compared to the potential chemical exposures. The physical hazards may include:

- Slips, trips, and falls;
- Material handling/back injury;
- Puncture wounds/cuts/abrasions.

All field team members are to be vigilant in providing clear footing, clearly identifying obstructions, holes, or other tripping hazards and maintaining an awareness of uneven terrain and slippery surfaces. During manual lifting tasks, all personnel will remember to lift with the force of the load suspended on their legs and not their backs. ECM does not expect employees to enter confined spaces during field activities. ECM does not expect any employees to enter a trench.

### **4.2 PHYSIOLOGICAL HAZARDS**

#### **4.2.1 Heat Stress**

Heat stress is caused by a number of interacting factors, including environmental conditions, clothing, workload, and the individual characteristics of the worker. Because heat stress is probably one of the most common (and potentially serious) illnesses, regular monitoring and other preventive precautions are vital.

ECM will monitor and address heat-related conditions as detailed in **Section 4.7.2.1**.

#### **4.2.2 Cold Stress**

Cold stress is caused by working in temperatures at or below freezing and/or in low wind chill conditions. During prolonged outdoor periods with inadequate clothing, effects of cold exposure may even occur at temperatures above freezing. Cold related emergencies can be the result of local (frostbite) or general (hypothermia) cooling of the body. Areas most commonly affected by frostbite are the ears, nose, hands, and feet. Lack of proper treatment can result in permanent damage to the affected body part. Hypothermia occurs when the body is unable to maintain its proper core (internal) temperature. If the person's condition is allowed to deteriorate, hypothermia will lead to death.

ECM will monitor and address cold-related conditions as detailed in **Section 4.7.2.2**.

#### **4.2.3 Altitude Sickness**

Altitude sickness can occur when a worker cannot get enough oxygen from the air at high altitudes. Air density, the number of molecules of both oxygen and nitrogen per given volume, drops as altitude increases. Consequently, the available amount of oxygen to sustain mental and physical alertness decreases with altitude. This may cause symptoms such as a headache, loss of appetite, dizziness,

muscle weakness and nausea. It happens most often when people who are not used to high altitudes go quickly from lower altitudes to 8000 feet or higher. The DEPO site elevation is approximately 7,560 feet, which is within range of potential hazard for those coming to the site from sea level elevation.

ECM will monitor and address altitude-related symptoms in workers. If mild symptoms occur, affected workers will rest at the current altitude to allow their bodies to adjust. If symptoms are severe, worker will be taken to nearest medical treatment center (**Appendix D**).

### 4.3 METEOROLOGICAL HAZARDS

Field team members must be aware of meteorological hazards and seek to minimize accidents and injuries that may occur during normal daily activities under adverse conditions such as hot or cold weather, thunderstorms, or other inclement weather events.

Employees must remain aware of changes in meteorologic conditions, such as changes to weather and forecast, prevailing wind direction, precipitation levels, or temperature fluctuations. ECM employees **will not participate in field activities in the event of an imminent lightning storm**. If thunder and lightning are observed, employees must act immediately:

- Seek shelter in an enclosed building.
- If no buildings are close by, get into a vehicle with the windows closed all the way.
- If out in open range with no shelter available, employees will squat down with feet together and only feet touching the ground. Cover ears (to protect against noise). Do not lie flat on the ground, as this avoids providing more surface area for a ground strike to affect.

Activities will be suspended until the storm has passed a safe distance beyond the work site and employees will seek shelter immediately. Work will recommence a half-hour after the lightning and thunder have ended.

ECM will monitor weather conditions in accordance with conditions as described in **Section 4.7.2.3**.

### 4.4 BIOLOGICAL HAZARDS

Insects, birds, snakes, reptiles, and small and large animals may be encountered. Risks include blood-borne pathogens (such as streptococcus infections from stings or tetanus infection from punctures), venomous injuries (such as bites/stings from spiders, insects or rattlesnake), allergic reactions (such as minor swelling or anaphylactic shock), and nuisance symptoms (such as swelling, itching, and pain). Bears are expected and likely to be present at or near the project site. It is recommended that all contact with bears be avoided. Proper food storage is mandatory. Do not leave food or food wrappers locked in vehicles. Promptly dispose of all waste (including food) in designated areas. Mountain lions (cougars) are also present in the area, but are rarely encountered. If you do encounter a lion, do not run. Talk calmly, avert your gaze, stand tall, and back away. Lions are primarily nocturnal, but they have attacked in broad daylight. Report all mountain lion encounters immediately.

Animal bites or stings are usually nuisances (localized swelling, itching, and minor pain) that can be handled by first aid treatments. The bites of certain snakes, lizards, spiders, and scorpions contain sufficient poison to warrant medical attention. While at the Site, ECM employees must remain vigilant and use particular caution:

- Avoid stepping into areas where you cannot clearly see the ground surface or reaching into areas that you cannot clearly see. Animals prefer dark, protected areas such as rock piles or beneath shrubs.
- Do not touch spiders or snakes if they are discovered. Wolf spiders are present and although their bite is not lethal, it is painful. Although uncommon, the Western rattlesnake is present.
- If bitten by a venomous snake or spider, contact 911 immediately.

- The injury should be iced pending paramedic arrival or transport to an emergency treatment facility.

There are diseases that can be transmitted by insect and animal bites. The greatest hazard and most common fatalities from animal bites, particularly bees, wasps, and spiders, are from a sensitivity reaction. Anaphylactic shock due to stings can lead to severe reactions in the circulatory, respiratory, and central nervous systems, which also can lead to death. Anyone assigned to work at the Site that is allergic will be required to carry a prescribed treatment kit, and the SSHO is to be told who is allergic. All stings or bites will be taken seriously. Anyone stung or bitten will be required to stop work while that person is observed for signs of severe swelling, shortness of breath, nausea, or shock. If there is any doubt, medical attention will be obtained.

Deer mice are possible carriers of Hantavirus. The most likely source of infection is from rodent urine and droppings inhaled as aerosols or dust. Initial symptoms are almost identical to the onset of flu. If you have potentially been exposed and exhibit flu-like symptoms, you should seek medical care immediately. Avoid rodent infested areas.

#### 4.5 CHEMICAL HAZARDS

There are potential chemical hazards from onsite contaminated media and the overall threat from exposure to these chemicals is being assessed by the NPS through the EE/CA process. However, minimal threat is anticipated during EE/CA Site Investigation activities because:

- (1) actual time onsite is short (minutes or up to a couple of hours)
- (2) minimal disturbance of, and exposure to, fine-grained material, is anticipated.

Normal safety precautions will be taken to minimize exposure to dust and fine-grained material that may be impacted with lead. Specific information on potential chemical hazards and their exposure characteristics are listed in the table below.

**Table 2: Chemical Hazards and Exposure Characteristics**

Chemical and Description	Exposure Limits and IDLH Level	Exposure Routes	Toxic Characteristics
Lead (dust)	PEL = 0.050 mg/m <sup>3</sup> IDLH = 100 mg/m <sup>3</sup> (NIOSH)	Inhalation, ingestion, skin and/or eye contact	Weakness, exhaustion, insomnia, facial pallor, anorexia, weight loss, malnutrition, constipation, abdominal pain, colic, anemia, gingival lead line, tremor, paralysis wrist/ankles, kidney disease, eye irritation, hypotension
<p>Notes:</p> <p>ACGIH American Conference of Governmental Industrial Hygienists (2004 TLVs)</p> <p>NIOSH National Institute for Occupational Safety and Health (August 2006 Pocket Guide)</p> <p>IDLH Immediately Dangerous to Life or Health</p> <p>PEL Permissible Exposure Limit</p> <p>mg/m<sup>3</sup> Milligram per cubic meter</p>			

**Appendix A** contains Material Safety Data Sheets (MSDS) for hazardous materials that may be brought to or present at the Site, such as sample preservatives, laboratory reagents and decontamination solutions. These materials are as follows:

- Nitric Acid (HNO<sub>3</sub>)
- Liquinox

#### 4.6 JOB HAZARD ANALYSIS

The purpose of a job hazard analysis (JHA) is to identify and quantify the health and safety hazards associated with each task and operation, and to evaluate the risk(s) to workers. Using this information, appropriate control methods are selected to eliminate the identified risks if possible, or to effectively control them. The control methods are documented in each task-specific JHA.

Each JHA lists the chemical hazards associated with that task and their known or anticipated airborne exposure during performance of the task. Each JHA also identifies anticipated physical and biological hazards and potential exposure levels or the likelihood of exposure. The final section of each JHA lists the control measures implemented to protect employees from exposure to the identified hazards. The JHA for each task summarized below is presented in **Appendix B**. The information provided here is designed to satisfy the job hazard analysis requirements of 1910.120(b)(4)(ii)(A) and the workplace hazard assessment requirements of 1910.132(d).

**Table 3: Job Hazard Analysis**

Task	Location	Hazard	JHA Control Measures
Mobilization	All locations	Slip, trip, fall;	Follow trails and cleared areas as much as possible, wear good hiking boots and always travel with partner
		Many areas of the park have vehicle traffic; vehicle can get stuck off-road;	Wear high visibility vest in high traffic areas; plan trip carefully to avoid hazardous driving areas. Walk an area prior to driving if conditions appear questionable.
Surface soil sampling	Background and suspect areas	Chemical exposure;	Level D PPE including nitrile sampling gloves. Obtain samples without creating airborne dust

Follow the safe work practices as outlined in the JHAs in **Appendix B** for each of the above during this project. The information in the JHAs and the attached MSDS (**Appendix A**) is made available to all employees who could be affected by it prior to the time they begin their work activities. Modifications to JHAs and the accompanying data sheets are communicated during routine briefings. ECM will inform other contractors and subcontractors about the nature and level of hazardous substances at this Site, and the likely degree of exposure to workers who participate in the project.

## **4.7 EXPOSURE MONITORING**

Field personnel will be responsible for completing exposure monitoring during field operations, as Site conditions warrant.

### **4.7.1 Airborne Dust Monitoring**

Given the Site conditions and planned field activities, dust is not a likely hazard. However, there is a potential for skin and/or inhalation exposure to airborne particles at the Site. ECM will visually monitor airborne dust within the worker's breathing zone during all tasks involving soil disturbances. Normal safety precautions will be taken to minimize exposure to dust and fine-grained material; however, ECM does not propose quantitative dust monitoring at this time.

### **4.7.2 Thermal Stress and Severe Weather**

#### **4.7.2.1 Heat Stress**

The implementation of preventative measures is the most effective way to limit the effects of heat-related illnesses. During periods of high heat, adequate liquids must be provided to replace lost body fluids. Replacement fluids can be a first aid electrolyte replacement solution, a commercial mix such as Gatorade, or a combination of these with fresh water. The replacement fluid temperature should be kept cool, 50° F to 60° F, and should be placed close to the work area. Employees must be encouraged to drink more than the amount required to satisfy thirst. Employees should also be encouraged to salt their foods more heavily during hot times of the year. Sunscreen or sunblock should be applied to exposed skin when working in direct sunlight over extended periods. In high heat personnel should stay out of direct sun when possible. Also, work may be scheduled to avoid the hottest times of the day.

Cooling devices such as vortex tubes or cooling vests can be worn beneath impermeable clothing. If cooling devices are worn, only physiological monitoring will be used to determine work activity. All workers are to rest when any symptoms of heat stress are noticed. Rest breaks are to be taken in a cool, shaded rest area.

Monitoring and emergency care procedures are provided in **Appendix C**.

#### **4.7.2.2 Cold Stress**

Persons working outdoors in temperatures at or below freezing and/or in low wind chill conditions may suffer from cold exposure. During prolonged outdoor periods with inadequate clothing, effects of cold exposure may even occur at temperatures above freezing. Cold related emergencies can be the result of local (frostbite) or general (hypothermia) cooling of the body. Areas most commonly affected by frostbite are the ears, nose, hands, and feet. Lack of proper treatment can result in permanent damage to the affected body part. Hypothermia occurs when the body is unable to maintain its proper core (internal) temperature. If the person's condition is allowed to deteriorate, hypothermia will lead to death.

Monitoring and emergency care procedures are provided in **Appendix C**.

#### **4.7.2.3 Severe Weather**

A means of obtaining real-time weather reports for local conditions must be maintained during all Site operations. ECM field personnel are responsible for monitoring and communicating weather conditions and ensuring that an appropriate rally point is established at each specific location visited at the Site.

##### **4.7.2.3.1 Lightning**

If a lightning storm is suspected or observed, all Site activities must be stopped, and Site equipment must be evaluated for its potential for acting as a lightning rod. Drill rig masts provide conduits for lightning to strike and injure workers. Personnel should wait indoors for the storm or lightning event to

end. If the strike of lightning occurs and personnel are out in the field, the response should be to disband from one another and lay low to the ground by dropping to your knees and bending forward with your hands wrapped around your knees, away from any poles or trees.

Persons struck by lightning receive a severe electrical shock and may be burned, but they carry no electrical charge and can be handled safely. Someone who appears to have been killed by lightning often can be revived by prompt action. Those unconscious but breathing probably will recover spontaneously. First aid and cardiopulmonary resuscitation (CPR) should be administered as appropriate until medical assistance arrives. Realize that victims who appear to be only stunned or otherwise unhurt also need attention. Check for burns, especially at fingers and toes and next to metal buckles, jewelry, or personal items that the victim is wearing. Remember to treat for shock.

#### 4.7.2.3.2 High Winds

If high winds are expected, or are encountered during work activities, appropriate action must be taken to ensure the protection of field personnel. If the winds present a hazard to personnel, field activities must be suspended until the storm passes.

#### 4.7.2.3.3 Flash Flooding

Flooding resulting from a thunderstorm presents a significant safety hazard, and must be continually monitored if a severe weather event is expected. If flooding presents a hazard to personnel at a specific location at the Site (e.g.: in vicinity of steep terrain) field activities must be temporarily suspended until the hazards are abated. **Do not attempt to cross a flooded stream or road in a vehicle or on foot. Do not enter any stream, if storms are apparent upstream.**

#### 4.7.2.3.4 Seasonal Weather Extremes

The weather in the mountains can change dramatically. Carry rain gear and warm clothing including wool socks, gloves and hat.

### 4.8 SITE CONTROL

The field team will control access to the site. Work zones will be established in order to delineate high-traffic locations, identify hazardous locations, and contain contamination within the smallest area possible. Employees entering the work zone must wear the proper personnel protective equipment (PPE; discussed in **Section 4.10**) for that area. Work and support areas will be established based on ambient air data, necessary security measures, and site-specific conditions. Only persons meeting the training and medical monitoring requirements and possessing proper PPE may enter the decontamination or work zones. Prior to start of the fieldwork, the SSHO shall undertake the following steps to secure the site:

Place flags, barricades, stakes, cones, tape, and/or lights to mark the site boundary and work zones and help prevent entry to the site by unauthorized personnel/vehicles.

Implement proper site communications to notify emergency response authorities and use appropriate communication devices when working in remote or restricted access areas. **Section 4.8.3** and **Section 5.1.1** include emergency contact telephone numbers.

#### 4.8.1 Site Map

**Figure 1** shows the DEPO Site.

#### 4.8.2 Buddy System

While working at DEPO, two ECM employees will be working together. In addition, an NPS employee may escort ECM into and out of each Site. The buddy system means that personnel work in pairs and stay in close visual contact to be able to observe one another and summon rapid assistance in case of an emergency. The responsibilities of workers using the buddy system include:

- Remaining in close visual contact with partner,
- Providing partner with assistance as needed or requested,
- Observing partner for signs of heat stress or other difficulties, and
- Notifying the Site manager or other Site personnel if emergency assistance is needed.

#### **4.8.3 Site Communications**

The following communication equipment may be employed:

- Cellular Phones: On ECM Personnel
- Two-way radios: On NPS Personnel
- Landlines: In case of emergency, call (760) 934-2289, or call 911. Cell phones work in some parts of recreation area, and there are pay phones at the marinas.
- Additional landlines include:
  - Inyo National Forest in Bishop, CA: (760) 873-2538

#### **4.9 TRAINING PROGRAM**

The training program is designed to ensure that workers receive the training they need to work safely on this Site. Site safety and health training requirements are based on the job hazard assessments (JHAs) and relevant OSHA requirements. ECM company employees generally have 40-hour HAZWOPER training and yearly refresher courses. Many employees have first aid and/or CPR training.

No other special training is required for the DEPO soil sampling.

#### **4.10 PERSONAL PROTECTIVE EQUIPMENT**

Personal protective equipment (PPE) assigned for this project is used to protect against employee exposures to hazardous substances and hazardous conditions as outlined in the JHA for this project.

Employees should use Level D protection during tasks:

- Long sleeve shirt and pants,
- Leather boots that cover the ankles and are sufficient for hiking several miles if necessary,
- Leather gloves/nitrile gloves as the task dictates, and
- Safety glasses during sampling activities

Level D is determined to be sufficient because there are no known or suspected hazardous substances at concentrations that meet or exceed the published exposure limits based on previous work conducted at the Site. Time at each of these sites with potentially impacted soil will be minimal and efforts will be made to minimize creation of dust.

#### **4.11 DECONTAMINATION**

Disposable sampling equipment will be used whenever possible to reduce equipment decontamination and expedite sampling. Decontamination will be required for a shovel, hand trowel, or any other non-disposable implement used to collect samples. The site-specific *Sampling and Analysis Plan* will provide equipment decontamination procedures in detail.

Prior to eating, drinking, or any other hand to mouth activity in the field, field personnel should wipe both hands and face using a damp cloth. At the end of the day, an effort should be made to brush dust off clothes and boots and wash hands and face thoroughly before eating a meal.



## **4.12 MEDICAL SURVEILLANCE**

Medical surveillance is used when there is the potential for worker exposure to hazardous substance at levels above OSHA permissible exposure limits or other published limits. The purpose of a medical surveillance program is to medically monitor worker health to ensure that personnel are not adversely affected by Site hazards. The provisions for medical surveillance are based on the Site characterization and job hazard analysis found in **Section 4** (above) and are consistent with OSHA requirements in 29 CFR 1910.120(f) and the following substance-specific requirements: *Lead* (1910.1025).

### **4.12.1 Medical Surveillance Program**

Medical surveillance requirements are based on a worker's potential for exposure as determined by the Site characterization and job hazard analysis documented in this HASP and on compliance with the requirements of 29 CFR 1910.120(f)(2). The ECM medical surveillance program is consistent with 29 CFR 1910.120(f) and addresses the following:

- Provisions of the Site medical surveillance program;
- Communication between the Site, physicians, and workers; and
- Medical recordkeeping procedures;

Based on documented exposure levels below permissible exposure limits, limited use of respirators (less than 30 days per year), and the absence of an employee-staffed HAZMAT team, a limited medical surveillance program is required and implemented at this Site. The person with responsibility for ensuring the ECM program is implemented and maintained is Andrew Campbell.

### **4.12.2 Medical Recordkeeping Procedures**

ECM's corporate medical recordkeeping procedures are consistent with the requirements of 29 CFR 1910.1020. A copy of the ECM medical monitoring program is available.

## **5. EMERGENCY RESPONSE PROCEDURES**

In the event of a catastrophe (fire, explosion, chemical release) or severe (life-threatening) medical emergency, field personnel will implement emergency response procedures. These procedures will enable field personnel involved in the emergency to respond appropriately. The procedures also establish the means of alerting responsible personnel to the emergency situation.

### **5.1 PERSONNEL INJURY AND MEDICAL EMERGENCIES**

Each vehicle will carry a basic first aid kit. The Site Supervisor and field personnel will possess knowledge of basic first aid. Staff will be first-aid trained by a recognized agency such as Red Cross.

1. Survey the situation:
  - Do not enter an area that may jeopardize your safety.
  - Prevent further injury.
  - Establish the patient's level of consciousness.
  - Call for help.
  - Contact emergency medical services and inform them of patient's condition.
2. Primary Assessment (patient unconscious)
  - Arousal
  - Airway
  - Breathing

- Circulation

**Only trained personnel should perform CPR or First Aid.**

3. Secondary Assessment (patient conscious)

- Check for bleeding: control with direct pressure.
- Do not move patient (unless location is not secure).
- Monitor vital signs.
- Provide First Aid to the level of your training.
- Contact the SSHO as soon as possible.
- Document the incident on Supervisor's Employee Injury Report (SEIR) form.

**5.1.1 Emergency Medical Assistance**

**In case of emergency:**

- Contact the nearest Ranger Station or Park Ranger, or
- Telephone the National Park Service at (760) 934-2289.

The nearest emergency medical facility is:

Mammoth Hospital  
85 Sierra Park Road  
Mammoth Lakes, California  
Switchboard: (760) 934-3311  
Emergency Services: (760) 924-4076

Hospital route maps are provided in **Appendix D**.

**5.2 FIRE HAZARDS**

Any area with dry vegetation is susceptible to fire danger. Catalytic converters on automobiles can contact dry grass, causing it to smolder and ignite. Other ignition sources are similarly dangerous.

ECM will not allow smoking or open flames on Site. ECM will select areas relatively free of grass and weeds for parking areas and then inspect the undersides of all vehicles for any vegetation contacting the vehicle's exhaust system. Any vegetation contacting the exhaust system will be removed by hand. A fire extinguisher (ABC, portable) will be in all field vehicles used during this project.

**5.3 REPORTING**

All emergency situations require follow-up and reporting. A verbal report of the incident must be submitted to the Program Manager within 24 hours. The report must include steps planned to prevent similar incidents from occurring. A report will be made to the Ranger Division through an NPS contact.

Reporting of spills or other environmental releases of reportable quantities of hazardous substances will be conducted in accordance of CERCLA reporting requirements.

The SSHO and the Site Supervisor should be involved in the corrective action and follow-up process to ensure its implementation at the Site.

## **Appendices**

Appendix A: Material Safety Data Sheets

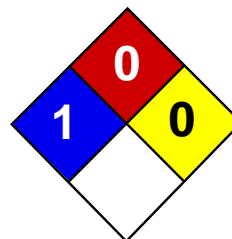
Appendix B: Job Hazard Analysis Sheets

Appendix C: Exposure Monitoring for Thermal Stress

Appendix D: Hospital Route Maps

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## **Appendix A: Material Safety Data Sheets**



Health	1
Fire	0
Reactivity	0
Personal Protection	E

## Material Safety Data Sheet

### Lead MSDS

#### Section 1: Chemical Product and Company Identification

**Product Name:** Lead

**Catalog Codes:** SLL1291, SLL1669, SLL1081, SLL1459, SLL1834

**CAS#:** 7439-92-1

**RTECS:** OF7525000

**TSCA:** TSCA 8(b) inventory: Lead

**CI#:** Not available.

**Synonym:** Lead Metal, granular; Lead Metal, foil; Lead Metal, sheet; Lead Metal, shot

**Chemical Name:** Lead

**Chemical Formula:** Pb

#### Contact Information:

**Sciencelab.com, Inc.**

14025 Smith Rd.

Houston, Texas 77396

US Sales: **1-800-901-7247**

International Sales: **1-281-441-4400**

Order Online: [ScienceLab.com](http://ScienceLab.com)

**CHEMTREC (24HR Emergency Telephone), call:**

1-800-424-9300

**International CHEMTREC, call:** 1-703-527-3887

**For non-emergency assistance, call:** 1-281-441-4400

#### Section 2: Composition and Information on Ingredients

##### Composition:

Name	CAS #	% by Weight
Lead	7439-92-1	100

**Toxicological Data on Ingredients:** Lead LD50: Not available. LC50: Not available.

#### Section 3: Hazards Identification

**Potential Acute Health Effects:** Slightly hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.

##### Potential Chronic Health Effects:

Slightly hazardous in case of skin contact (permeator). CARCINOGENIC EFFECTS: Classified A3 (Proven for animal.) by ACGIH, 2B (Possible for human.) by IARC. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to blood, kidneys, central nervous system (CNS). Repeated or prolonged exposure to the substance can produce target organs damage.

#### Section 4: First Aid Measures

**Eye Contact:**

Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention if irritation occurs.

**Skin Contact:** Wash with soap and water. Cover the irritated skin with an emollient. Get medical attention if irritation develops.

**Serious Skin Contact:** Not available.

**Inhalation:**

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.

**Serious Inhalation:** Not available.

**Ingestion:**

Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. If large quantities of this material are swallowed, call a physician immediately. Loosen tight clothing such as a collar, tie, belt or waistband.

**Serious Ingestion:** Not available.

## Section 5: Fire and Explosion Data

**Flammability of the Product:** May be combustible at high temperature.

**Auto-Ignition Temperature:** Not available.

**Flash Points:** Not available.

**Flammable Limits:** Not available.

**Products of Combustion:** Some metallic oxides.

**Fire Hazards in Presence of Various Substances:** Non-flammable in presence of open flames and sparks, of shocks, of heat.

**Explosion Hazards in Presence of Various Substances:**

Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

**Fire Fighting Media and Instructions:**

SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray, fog or foam. Do not use water jet.

**Special Remarks on Fire Hazards:** When heated to decomposition it emits highly toxic fumes of lead.

**Special Remarks on Explosion Hazards:** Not available.

## Section 6: Accidental Release Measures

**Small Spill:**

Use appropriate tools to put the spilled solid in a convenient waste disposal container. Finish cleaning by spreading water on the contaminated surface and dispose of according to local and regional authority requirements.

**Large Spill:**

Use a shovel to put the material into a convenient waste disposal container. Finish cleaning by spreading water on the contaminated surface and allow to evacuate through the sanitary system. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

## Section 7: Handling and Storage

**Precautions:**

Keep locked up.. Keep away from heat. Keep away from sources of ignition. Empty containers pose a fire risk, evaporate the residue under a fume hood. Ground all equipment containing material. Do not ingest. Do not breathe dust. Wear suitable

protective clothing. If ingested, seek medical advice immediately and show the container or the label. Keep away from incompatibles such as oxidizing agents.

**Storage:** Keep container tightly closed. Keep container in a cool, well-ventilated area.

## Section 8: Exposure Controls/Personal Protection

### Engineering Controls:

Use process enclosures, local exhaust ventilation, or other engineering controls to keep airborne levels below recommended exposure limits. If user operations generate dust, fume or mist, use ventilation to keep exposure to airborne contaminants below the exposure limit.

**Personal Protection:** Safety glasses. Lab coat. Dust respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

### Personal Protection in Case of a Large Spill:

Splash goggles. Full suit. Dust respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

### Exposure Limits:

TWA: 0.05 (mg/m<sup>3</sup>) from ACGIH (TLV) [United States] TWA: 0.05 (mg/m<sup>3</sup>) from OSHA (PEL) [United States] TWA: 0.03 (mg/m<sup>3</sup>) from NIOSH [United States] TWA: 0.05 (mg/m<sup>3</sup>) [Canada] Consult local authorities for acceptable exposure limits.

## Section 9: Physical and Chemical Properties

**Physical state and appearance:** Solid. (Metal solid.)

**Odor:** Not available.

**Taste:** Not available.

**Molecular Weight:** 207.21 g/mole

**Color:** Bluish-white. Silvery. Gray

**pH (1% soln/water):** Not applicable.

**Boiling Point:** 1740°C (3164°F)

**Melting Point:** 327.43°C (621.4°F)

**Critical Temperature:** Not available.

**Specific Gravity:** 11.3 (Water = 1)

**Vapor Pressure:** Not applicable.

**Vapor Density:** Not available.

**Volatility:** Not available.

**Odor Threshold:** Not available.

**Water/Oil Dist. Coeff.:** Not available.

**Ionicity (in Water):** Not available.

**Dispersion Properties:** Not available.

**Solubility:** Insoluble in cold water.

## Section 10: Stability and Reactivity Data

**Stability:** The product is stable.

**Instability Temperature:** Not available.

**Conditions of Instability:** Incompatible materials, excess heat

**Incompatibility with various substances:** Reactive with oxidizing agents.

**Corrosivity:** Non-corrosive in presence of glass.

**Special Remarks on Reactivity:**

Can react vigorously with oxidizing materials. Incompatible with sodium carbide, chlorine trifluoride, trioxane + hydrogen peroxide, ammonium nitrate, sodium azide, disodium acetylide, sodium acetylide, hot concentrated nitric acid, hot concentrated hydrochloric acid, hot concentrated sulfuric acid, zirconium.

**Special Remarks on Corrosivity:** Not available.

**Polymerization:** Will not occur.

## Section 11: Toxicological Information

**Routes of Entry:** Absorbed through skin. Inhalation. Ingestion.

**Toxicity to Animals:**

LD50: Not available. LC50: Not available.

**Chronic Effects on Humans:**

CARCINOGENIC EFFECTS: Classified A3 (Proven for animal.) by ACGIH, 2B (Possible for human.) by IARC. May cause damage to the following organs: blood, kidneys, central nervous system (CNS).

**Other Toxic Effects on Humans:** Slightly hazardous in case of skin contact (irritant), of ingestion, of inhalation.

**Special Remarks on Toxicity to Animals:** Not available.

**Special Remarks on Chronic Effects on Humans:** Not available.

**Special Remarks on other Toxic Effects on Humans:**

Acute Potential: Skin: Lead metal granules or dust: May cause skin irritation by mechanical action. Lead metal foil, shot or sheets: Not likely to cause skin irritation Eyes: Lead metal granules or dust: Can irritate eyes by mechanical action. Lead metal foil, shot or sheets: No hazard. Will not cause eye irritation. Inhalation: In an industrial setting, exposure to lead mainly occurs from inhalation of dust or fumes. Lead dust or fumes: Can irritate the upper respiratory tract (nose, throat) as well as the bronchi and lungs by mechanical action. Lead dust can be absorbed through the respiratory system. However, inhaled lead does not accumulate in the lungs. All of an inhaled dose is eventually absorbed or transferred to the gastrointestinal tract. Inhalation effects of exposure to fumes or dust of inorganic lead may not develop quickly. Symptoms may include metallic taste, chest pain, decreased physical fitness, fatigue, sleep disturbance, headache, irritability, reduces memory, mood and personality changes, aching bones and muscles, constipation, abdominal pains, decreasing appetite. Inhalation of large amounts may lead to ataxia, delirium, convulsions/seizures, coma, and death. Lead metal foil, shot, or sheets: Not an inhalation hazard unless metal is heated. If metal is heated, fumes will be released. Inhalation of these fumes may cause "fume metal fever", which is characterized by flu-like symptoms. Symptoms may include metallic taste, fever, nausea, vomiting, chills, cough, weakness, chest pain, generalized muscle pain/aches, and increased white blood cell count. Ingestion: Lead metal granules or dust: The symptoms of lead poisoning include abdominal pain or cramps (lead colic), spasms, nausea, vomiting, headache, muscle weakness, hallucinations, distorted perceptions, "lead line" on the gums, metallic taste, loss of appetite, insomnia, dizziness and other symptoms similar to that of inhalation. Acute poisoning may result in high lead levels in the blood and urine, shock, coma and death in extreme cases. Lead metal foil, shot or sheets: Not an ingestion hazard for usual industrial handling.

## Section 12: Ecological Information

**Ecotoxicity:** Not available.

**BOD5 and COD:** Not available.



**Products of Biodegradation:**

Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

**Toxicity of the Products of Biodegradation:** The products of degradation are less toxic than the product itself.

**Special Remarks on the Products of Biodegradation:** Not available.

**Section 13: Disposal Considerations****Waste Disposal:**

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

**Section 14: Transport Information**

**DOT Classification:** Not a DOT controlled material (United States).

**Identification:** Not applicable.

**Special Provisions for Transport:** Not applicable.

**Section 15: Other Regulatory Information****Federal and State Regulations:**

California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Lead California prop. 65: This product contains the following ingredients for which the State of California has found to cause reproductive harm (female) which would require a warning under the statute: Lead California prop. 65: This product contains the following ingredients for which the State of California has found to cause reproductive harm (male) which would require a warning under the statute: Lead California prop. 65 (no significant risk level): Lead: 0.0005 mg/day (value) California prop. 65: This product contains the following ingredients for which the State of California has found to cause birth defects which would require a warning under the statute: Lead California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: Lead Connecticut hazardous material survey.: Lead Illinois toxic substances disclosure to employee act: Lead Illinois chemical safety act: Lead New York release reporting list: Lead Rhode Island RTK hazardous substances: Lead Pennsylvania RTK: Lead

**Other Regulations:**

OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

**Other Classifications:**

**WHMIS (Canada):** CLASS D-2A: Material causing other toxic effects (VERY TOXIC).

**DSCL (EEC):**

R20/22- Harmful by inhalation and if swallowed. R33- Danger of cumulative effects. R61- May cause harm to the unborn child. R62- Possible risk of impaired fertility. S36/37- Wear suitable protective clothing and gloves. S44- If you feel unwell, seek medical advice (show the label when possible). S53- Avoid exposure - obtain special instructions before use.

**HMIS (U.S.A.):**

**Health Hazard:** 1

**Fire Hazard:** 0

**Reactivity:** 0

**Personal Protection:** E

**National Fire Protection Association (U.S.A.):**

**Health:** 1

**Flammability:** 0

**Reactivity:** 0

**Specific hazard:**

**Protective Equipment:**

Gloves. Lab coat. Dust respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Safety glasses.

## Section 16: Other Information

**References:** Not available.

**Other Special Considerations:** Not available.

**Created:** 10/10/2005 08:21 PM

**Last Updated:** 06/09/2012 12:00 PM

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## LIQUINOX MSDS

### Section 1 : PRODUCT AND COMPANY IDENTIFICATION

**Chemical family:** Detergent.

**Manufacturer:** Alconox, Inc.  
30 Glenn St.  
Suite 309  
White Plains, NY 10603.

**Manufacturer emergency** 800-255-3924.

**phone number:** 813-248-0585 (outside of the United States).

**Supplier:** Same as manufacturer.

**Product name:** Liquinox

### Section 2 : INGREDIENT INFORMATION

C.A.S.	CONCENTRATION %	Ingredient Name	T.L.V.	LD/50	LC/50
25155-30-0	10-30	SODIUM DODECYLBENZENESULFONATE	NOT AVAILABLE	438 MG/KG RAT ORAL  1330 MG/KG MOUSE ORAL	NOT AVAILABLE

### Section 3 : HAZARD IDENTIFICATION

**Route of entry:** Skin contact, eye contact, inhalation and ingestion.

**Effects of acute exposure**

**Eye contact:** May cause irritation.

**Skin contact:** Prolonged and repeated contact may cause irritation.

**Inhalation:** May cause headache and nausea.

**Ingestion:** May cause vomiting and diarrhea.  
May cause gastric distress.

**Effects of chronic exposure:** See effects of acute exposure.

### Section 4 : FIRST AID MEASURES

**Skin contact:** Remove contaminated clothing.  
Wash thoroughly with soap and water.  
Seek medical attention if irritation persists.

**Eye contact:** Check for and remove contact lenses.  
Flush eyes with clear, running water for 15 minutes while holding eyelids open: if irritation persists, consult a physician.

**Inhalation:** Remove victim to fresh air.  
If irritation persists, seek medical attention.

**Ingestion:** Do not induce vomiting, seek medical attention.  
Dilute with two glasses of water.  
Never give anything by mouth to an unconscious person.

#### Section 5 : FIRE FIGHTING MEASURES

**Flammability:** Not flammable.

**Conditions of flammability:** Surrounding fire.

**Extinguishing media:** Carbon dioxide, dry chemical, foam.  
Water  
Water fog.

**Special procedures:** Self-contained breathing apparatus required.  
Firefighters should wear the usual protective gear.  
Use water spray to cool fire exposed containers.

**Auto-ignition temperature:** Not available.

**Flash point (°C), method:** None

**Lower flammability limit (% vol):** Not applicable.

**Upper flammability limit (% vol):** Not applicable.

**Explosion Data**

**Sensitivity to static discharge:** Not available.

**Sensitivity to mechanical impact:** Not available.

**Hazardous combustion products:** Oxides of carbon (COx).  
Hydrocarbons.

**Rate of burning:** Not available.

**Explosive power:** Containers may rupture if exposed to heat or fire.

#### Section 6 : ACCIDENTAL RELEASE MEASURES

**Leak/Spill:** Contain the spill.  
Prevent entry into drains, sewers, and other waterways.  
Wear appropriate protective equipment.  
Small amounts may be flushed to sewer with water.  
Soak up with an absorbent material.  
Place in appropriate container for disposal.  
Notify the appropriate authorities as required.

#### Section 7 : HANDLING AND STORAGE

**Handling procedures and equipment:** Protect against physical damage.  
Avoid breathing vapors/mists.  
Wear personal protective equipment appropriate to task.  
Wash thoroughly after handling.  
Keep out of reach of children.  
Avoid contact with skin, eyes and clothing.  
Avoid extreme temperatures.  
Launder contaminated clothing prior to reuse.

**Storage requirements:** Store away from incompatible materials.  
Keep containers closed when not in use.

## Section 8 : EXPOSURE CONTROLS / PERSONAL PROTECTION

### Precautionary Measures

**Gloves/Type:**



Wear appropriate gloves.

**Respiratory/Type:** None required under normal use.

**Eye/Type:**



Safety glasses recommended.

**Footwear/Type:** Safety shoes per local regulations.

**Clothing/Type:** As required to prevent skin contact.

**Other/Type:** Eye wash facility should be in close proximity.  
Emergency shower should be in close proximity.

**Ventilation requirements:** Local exhaust at points of emission.

**Exposure limit of material:** Not available.

## Section 9 : PHYSICAL AND CHEMICAL PROPERTIES

**Physical state:** Liquid.

**Appearance & odor:** Odourless.  
Pale yellow.

**Odor threshold (ppm):** Not available.

**Vapour pressure @ 20°C (68°F):**  
**(mmHg):** 17

**Vapour density (air=1):** >1

### Volatiles (%)

**By volume:** Not available.

**Evaporation rate (butyl acetate = 1):** < 1.

**Boiling point (°C):** 100 (212F)

**Freezing point (°C):** Not available.

**pH:** 8.5

**Specific gravity @ 20 °C:** (water = 1).  
1.083

**Solubility in water (%):** Complete.

**Coefficient of water\oil dist.:** Not available.

**VOC:** None

**Chemical family:** Detergent.

## Section 10 : STABILITY AND REACTIVITY

**Chemical stability:** Product is stable under normal handling and storage conditions.

**Conditions of instability:** Extreme temperatures.

**Hazardous polymerization:** Will not occur.

**Incompatible substances:** Strong acids.  
Strong oxidizing agents.

**Hazardous decomposition products:** See hazardous combustion products.

#### Section 11 : TOXICOLOGICAL INFORMATION

**LD50 of product, species & route:** > 5000 mg/kg rat oral.

**LC50 of product, species & route:** Not available.

**Sensitization to product:** Not available.

**Carcinogenic effects:** Not listed as a carcinogen.

**Reproductive effects:** Not available.

**Teratogenicity:** Not available.

**Mutagenicity:** Not available.

**Synergistic materials:** Not available.

#### Section 12 : ECOLOGICAL INFORMATION

**Environmental toxicity:** No data at this time.

**Environmental fate:** No data at this time.

#### Section 13 : DISPOSAL CONSIDERATIONS

**Waste disposal:** In accordance with local and federal regulations.

#### Section 14 : TRANSPORT INFORMATION

**D.O.T. CLASSIFICATION:** Not regulated.

**Special shipping information:** Not regulated.

#### Section 15 : REGULATORY INFORMATION

##### Canadian Regulatory Information

**WHMIS classification:** Not controlled.

**DSL status:** Not available.

##### USA Regulatory Information

**SARA hazard catagories sections 311/312:** Immediate (Acute) Health Hazard: No.  
Delayed (Chronic) Health Hazard: No.  
Fire Hazard: No.  
Sudden Release of Pressure: No.  
Reactive: No.

**SARA Section 313:** None

**TSCA inventory:** All components of this product are listed on the TSCA inventory.

**NFPA**

**Health Hazard:** 1

**Flammability:** 0

**Reactivity:** 0

**HMIS**

**Health Hazard:** 1

**Flammability:** 0

**Physical hazard:** 0

**PPE:** A

<b>Section 16 : OTHER INFORMATION</b>
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**Supplier MSDS date:** 2006/07/14

**Data prepared by:** Global Safety Management  
3340 Peachtree Road, #1800  
Atlanta, GA 30326

Phone: 877-683-7460

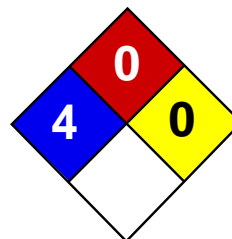
Fax: (877) 683-7462

Web: [www.globalsafetynet.com](http://www.globalsafetynet.com)

Email: [info@globalsafetynet.com](mailto:info@globalsafetynet.com).

**General note:** This material safety data sheet was prepared from information obtained from various sources, including product suppliers and the Canadian Center for Occupational Health and Safety.

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Health	3
Fire	0
Reactivity	0
Personal Protection	

## Material Safety Data Sheet

### Nitric acid, 65% MSDS

#### Section 1: Chemical Product and Company Identification

**Product Name:** Nitric acid, 65%

**Catalog Codes:** SLN2161

**CAS#:** Mixture.

**RTECS:** Not applicable.

**TSCA:** TSCA 8(b) inventory: Water; Nitric acid, fuming

**CI#:** Not applicable.

**Synonym:** Nitric Acid, 65%

**Chemical Name:** Not applicable.

**Chemical Formula:** Not applicable.

#### Contact Information:

**Sciencelab.com, Inc.**

14025 Smith Rd.

Houston, Texas 77396

US Sales: **1-800-901-7247**

International Sales: **1-281-441-4400**

Order Online: [ScienceLab.com](http://ScienceLab.com)

**CHEMTREC (24HR Emergency Telephone), call:**

1-800-424-9300

**International CHEMTREC, call:** 1-703-527-3887

**For non-emergency assistance, call:** 1-281-441-4400

#### Section 2: Composition and Information on Ingredients

##### Composition:

Name	CAS #	% by Weight
Water	7732-18-5	35
Nitric acid, fuming	7697-37-2	65

**Toxicological Data on Ingredients:** Nitric acid, fuming: VAPOR (LC50): Acute: 244 ppm 0.5 hours [Rat]. 344 ppm 0.5 hours [Rat].

#### Section 3: Hazards Identification

##### Potential Acute Health Effects:

Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, . Slightly hazardous in case of inhalation (lung sensitizer). Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Prolonged exposure may result in skin burns and ulcerations. Over-exposure by inhalation may cause respiratory irritation. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

##### Potential Chronic Health Effects:

CARCINOGENIC EFFECTS: Not available. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to lungs, mucous membranes, upper respiratory



tract, skin, eyes, teeth. Repeated or prolonged exposure to the substance can produce target organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection.

#### Section 4: First Aid Measures

**Eye Contact:**

Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

**Skin Contact:**

In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

**Serious Skin Contact:**

Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

**Inhalation:**

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

**Serious Inhalation:**

Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. **WARNING:** It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

**Ingestion:**

If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

**Serious Ingestion:** Not available.

#### Section 5: Fire and Explosion Data

**Flammability of the Product:** Non-flammable.

**Auto-Ignition Temperature:** Not applicable.

**Flash Points:** Not applicable.

**Flammable Limits:** Not applicable.

**Products of Combustion:** Not available.

**Fire Hazards in Presence of Various Substances:** of combustible materials

**Explosion Hazards in Presence of Various Substances:**

Explosive in presence of reducing materials, of organic materials, of metals, of alkalis. Non-explosive in presence of open flames and sparks, of shocks.

**Fire Fighting Media and Instructions:** Not applicable.

**Special Remarks on Fire Hazards:**

Flammable in presence of cellulose or other combustible materials. Phosphine, hydrogen sulfide, selenide all ignite when fuming nitric acid is dripped into gas. (Nitric Acid, fuming)

**Special Remarks on Explosion Hazards:**

Reacts explosively with metallic powders, carbides, cyanides, sulfides, alkalies and turpentine. Can react explosively with many reducing agents. Arsine, phosphine, tetraborane all oxidized explosively in presence of nitric acid. Cesium and rubidium

acetylides explode in contact with nitric acid. Explosive reaction with Nitric Acid + Nitrobenzene + water. Detonation with Nitric Acid + 4-Methylcyclohexane. (Nitric acid, fuming)

## Section 6: Accidental Release Measures

### Small Spill:

Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. If necessary: Neutralize the residue with a dilute solution of sodium carbonate.

### Large Spill:

Corrosive liquid. Oxidizing material. Poisonous liquid. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Avoid contact with a combustible material (wood, paper, oil, clothing...). Keep substance damp using water spray. Do not touch spilled material. Use water spray curtain to divert vapor drift. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Neutralize the residue with a dilute solution of sodium carbonate. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

## Section 7: Handling and Storage

### Precautions:

Keep locked up.. Keep container dry. Keep away from heat. Keep away from sources of ignition. Keep away from combustible material.. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as reducing agents, combustible materials, organic materials, metals, acids, alkalis, moisture. May corrode metallic surfaces. Store in a metallic or coated fiberboard drum using a strong polyethylene inner package.

### Storage:

Keep container tightly closed. Keep container in a cool, well-ventilated area. Separate from acids, alkalies, reducing agents and combustibles. See NFPA 43A, Code for the Storage of Liquid and Solid Oxidizers. Do not store above 23°C (73.4°F).

## Section 8: Exposure Controls/Personal Protection

### Engineering Controls:

Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

### Personal Protection:

Face shield. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves. Boots.

### Personal Protection in Case of a Large Spill:

Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

### Exposure Limits:

TWA: 2 STEL: 4 (ppm) from ACGIH (TLV) [United States] TWA: 2 STEL: 4 from OSHA (PEL) [United States] Consult local authorities for acceptable exposure limits.

## Section 9: Physical and Chemical Properties

**Physical state and appearance:** Liquid.

**Odor:** Acrid. Disagreeable and choking. (Strong.)

**Taste:** Not available.

**Molecular Weight:** Not applicable.

**Color:** Colorless to light yellow.

**pH (1% soln/water):** Acidic.

**Boiling Point:** 121°C (249.8°F)

**Melting Point:** -41.6°C (-42.9°F)

**Critical Temperature:** Not available.

**Specific Gravity:** 1.408 (Water = 1)

**Vapor Pressure:** 6 kPa (@ 20°C)

**Vapor Density:** 2.5 (Air = 1)

**Volatility:** Not available.

**Odor Threshold:** 0.29 ppm

**Water/Oil Dist. Coeff.:** Not available.

**Ionicity (in Water):** Not available.

**Dispersion Properties:** See solubility in water, diethyl ether.

**Solubility:**  
Easily soluble in cold water, hot water. Soluble in diethyl ether.

## Section 10: Stability and Reactivity Data

**Stability:** The product is stable.

**Instability Temperature:** Not available.

**Conditions of Instability:** Incompatible materials

**Incompatibility with various substances:**  
Highly reactive with alkalis. Reactive with reducing agents, combustible materials, organic materials, metals, acids.

**Corrosivity:**  
Extremely corrosive in presence of aluminum, of copper. Non-corrosive in presence of glass, of stainless steel(304), of stainless steel(316), of brass.

**Special Remarks on Reactivity:**  
A strong oxidizer. Reacts violently with alcohol, organic material, turpene, charcoal. Violent reaction with Nitric acid + Acetone and Sulfuric acid. Nitric Acid will react with water or steam to produce heat and toxic, corrosive and flammable vapors. (Nitric acid, fuming)

**Special Remarks on Corrosivity:**  
In presence of traces of oxides, it attacks all base metals except aluminum and special chromium steels. It will attack some forms of plastics, rubber, and coatings. No corrosive effect on bronze. No corrosivity data for zinc, and steel

**Polymerization:** Will not occur.

## Section 11: Toxicological Information

**Routes of Entry:** Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

**Toxicity to Animals:**  
LD50: Not available. LC50: Not available.

**Chronic Effects on Humans:**

Contains material which may cause damage to the following organs: lungs, mucous membranes, upper respiratory tract, skin, eyes, teeth.

**Other Toxic Effects on Humans:**

Extremely hazardous in case of inhalation (lung corrosive). Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (corrosive), of ingestion, .

**Special Remarks on Toxicity to Animals:** LDL - Lowest Published Lethal Dose [Human] - Route: Oral; Dose: 430 mg/kg (Nitric acid, fuming)

**Special Remarks on Chronic Effects on Humans:**

May cause adverse reproductive effects (effects on newborn and fetotoxicity) based on animal data. (Nitric acid, fuming)

**Special Remarks on other Toxic Effects on Humans:**

Acute Potential Health Effects: Skin: Severely irritates skin. Causes skin burns and may cause deep and penetrating ulcers of the skin with a characteristic yellow to brownish discoloration. May be fatal if absorbed through skin. Eyes: Severely irritates eyes. Causes eye burns. May cause irreversible eye injury. Ingestion: May be fatal if swallowed. Causes serious gastrointestinal tract irritation or burns with nausea, vomiting, severe abdominal pain, and possible "coffee grounds" appearance of the vomitus . May cause perforation of the digestive tract. Inhalation: May be fatal if inhaled. Vapor is extremely hazardous. Vapor may cause nitrous gas poisoning. Effects may be delayed. May cause irritation of the mucous membranes and respiratory tract with burning pain in the nose and throat, coughing, sneezing, wheezing, shortness of breath and pulmonary edema. Other symptoms may include nausea, and vomiting. Chronic Potential Health Effects: Repeated inhalation may produce changes in pulmonary function and/or chronic bronchitis. It may also affect behavior (headache, dizziness, drowsiness, muscle contraction or spasticity, weakness, loss of coordination, mental confusion), and urinary system (kidney failure, decreased urinary output after several hours of

## Section 12: Ecological Information

**Ecotoxicity:** Not available.

**BOD5 and COD:** Not available.

**Products of Biodegradation:**

Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

**Toxicity of the Products of Biodegradation:** The products of degradation are less toxic than the product itself.

**Special Remarks on the Products of Biodegradation:** Not available.

## Section 13: Disposal Considerations

**Waste Disposal:**

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

## Section 14: Transport Information

**DOT Classification:** Class 8: Corrosive material

**Identification:** : Nitric acid UNNA: 2031 PG: II

**Special Provisions for Transport:** Marine Pollutant

## Section 15: Other Regulatory Information

**Federal and State Regulations:**

New York release reporting list: Nitric acid, fuming Rhode Island RTK hazardous substances: Nitric acid, fuming Pennsylvania RTK: Nitric acid, fuming Florida: Nitric acid, fuming Minnesota: Nitric acid, fuming Massachusetts RTK: Nitric acid, fuming

New Jersey: Nitric acid, fuming TSCA 8(b) inventory: Water; Nitric acid, fuming SARA 302/304/311/312 extremely hazardous substances: Nitric acid, fuming SARA 313 toxic chemical notification and release reporting: Nitric acid, fuming 65% CERCLA: Hazardous substances.: Nitric acid, fuming: 1000 lbs. (453.6 kg);

**Other Regulations:** OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

**Other Classifications:**

**WHMIS (Canada):**

CLASS D-1A: Material causing immediate and serious toxic effects (VERY TOXIC). CLASS D-2A: Material causing other toxic effects (VERY TOXIC). CLASS E: Corrosive liquid.

**DSCL (EEC):**

R8- Contact with combustible material may cause fire. R35- Causes severe burns. S23- Do not breathe gas/fumes/vapour/spray [\*\*\*] S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S36- Wear suitable protective clothing. S45- In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

**HMIS (U.S.A.):**

**Health Hazard:** 3

**Fire Hazard:** 0

**Reactivity:** 0

**Personal Protection:**

**National Fire Protection Association (U.S.A.):**

**Health:** 4

**Flammability:** 0

**Reactivity:** 0

**Specific hazard:**

**Protective Equipment:**

Gloves. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Face shield.

## Section 16: Other Information

**References:** Not available.

**Other Special Considerations:** Not available.

**Created:** 10/10/2005 10:59 AM

**Last Updated:** 11/06/2008 12:00 PM

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## **Appendix B: Job Hazard Analysis Sheets**

## APPENDIX B: JOB HAZARD ANALYSIS

Task	Location	Hazard	JHA Control Measures
All tasks	Areas of tailings	Chemical exposure to metals	Dust suppression, avoid creating dust, avoid work during windy weather
All tasks	All areas	Heat/Cold stress	Buddy system personnel monitoring for symptoms of heat stress including cramps, faintness, dizziness or disorientation, and pale, clammy skin, work/rest regime, fluid intake
All tasks	All areas	Inclement weather	Stop work and shelter in electrical storms or other severe weather
All tasks	All areas	Remote location, Rough Terrain (slips, trips, falls)	Use extra caution on rough terrain, carefully plan activities to allow time to get in, do work and get out without rushing, work only during daylight hours, communicate location with "base camp". Cell phones available but reception can be intermittent.  ECM personnel will work in pairs.
All tasks	All areas	Biological hazards	Choose sampling locations carefully-watch for wasps, hornets, bees, spiders, and rattlesnakes. If you see a bear, make as much noise as possible by yelling very loudly. Store food and scented items in vehicle trunk or covered out of site with food sealed in air-tight containers in vehicles without trunks. Use bear canisters or boxes when not in your vehicle. Personnel to check for plant material and insects on clothing at the end of each workday. A standard first-aid kit will be available.

<b>Job Hazard Analysis - Driving</b>	
Field staff must review job-specific work plan and coordinate with project manager to verify that all up-front logistics are completed prior to starting work including, but not limited to, permitting, access agreements and notification to required contacts (e.g., Site managers, inspectors, clients, subcontractors, etc.). Additionally, a tailgate safety meeting must be performed and documented at the beginning of each work day. Also consider weather conditions (heat, cold, rain, lightning).	
<b>Date:</b>	March 2013
<b>JHA Type:</b>	
<b>Work Type:</b>	Driving - Personal, Rental or Company Vehicles
<b>Work Site:</b>	DEPO
<b>Organization:</b>	ECM
<b>Personal Protective Equipment (PPE) needed</b>	
First Aid Kit	
Other - Sunglasses and safety belts	

No	Job Steps	Potential Hazard(s)	Critical Action(s)
1	PRE-TRIP - Review JHA and project work plan	Consider worst case outcome of vehicle operation (blowout, breakdown, collision, injury or death, lost on journey)	Assess the potential hazards to performing this task. Analyze how to reduce the risks. Act to ensure safe operation of the vehicle. Review directions to planned location.
2	Perform perimeter walk around of vehicle for damage or unusual conditions.	Low air pressure, flat tire, blowout, impaired vision, collision, injury or death, vehicle is not adequate for trip.	Assure tires are properly inflated and there is sufficient tread (including spare). Assure there are no cuts or bulges in the sidewalls, all wheels/rims are in good condition. Assure windshield and window glass is clean and free from obstructions. Lift wiper arms and check wiper blades for damage or deterioration. Check to see that all lights work. Check for fluid leaks under vehicle. Check behind vehicle for obstructions.
3	Check and adjust seat steering wheel and headrest, mirrors. Check to see that headlamps turn signals, backup lights, hazard lights are working properly, washer/wipers.	Back or body strain. Blind spots. Inability to signal intentions, other vehicles cannot see you. Streaking windshield, impaired vision.	Adjust seat, steering wheel height and headrest so body is fully supported, upper arms close to body, pedals within easy reach. Lower steering wheel so hands are below shoulders and shoulders are relaxed. Check mirror adjustments each time vehicle is re-started. Test operations of headlights, front and rear turn signals, backup lights. Locate and test operation of headlamp, wiper and washer switches. Check oil, radiator, brake and washer fluid levels.
4	Fasten seat belts. Make sure passenger air bag is turned on.	Increased risk of more serious injury or death in collision. Ejection from vehicle in collision.	Assure seat belt is in good condition and fastened. Assure all passenger seat belts are in good condition, fastened and working. Turn on passenger air bag if necessary.
5	Lock doors.	Ejection from vehicle in collision. Unwanted intrusion.	Manually lock all doors to vehicle.
6	Start engine.	Unexpected movement.	Assure that transmission is in 'Park' and that parking brake is set.



No	Job Steps	Potential Hazard(s)	Critical Action(s)
7	Check gauges and warning lights while engine is warming up.	Overheated engine or breakdown due to lack of critical fluids. Brake failure. Stranding.	Assure there is sufficient gas, oil, and other critical fluids, by checking warning lights and previously checking fluid reservoirs. Turn on headlights.
8	Slowly pull out of parking space.	Collision with other vehicles, pedestrians, or stationary	Release parking brake. Check mirrors and over shoulder in all directions prior to slowly pulling out of parking space. Signal if parallel
9	HIGHWAY DRIVING - Keep your eyes moving.	Collision, injury or death to occupants or other parties.	DRIVE DEFENSIVELY. Move eyes at least every 2 seconds. Scan major and minor intersections before entry (left-right-left). Check mirrors when slowing or stopping vehicle. Scan mirrors frequently, at least one mirror every 5-8 seconds. Avoid staring while evaluating road conditions. Do not use cell phones or perform other distraction activities while car is in motion. If necessary, pull off the roadway and park prior to performing other activities.
10	Aim high in steering.	Collision, injury or death to occupants or other parties.	Maintain 15 second eye lead time (1 1/2 blocks in city traffic, 1/4 mile in highway traffic). Assess information from distant objects (i.e., flashers on?). Adjust eye lead distance to speed.
11	Leave yourself an out.	Collision, injury or death to occupants or other parties.	Maintain safety cushion around vehicle (front, sides, rear). Adjust vehicle space and speed to avoid unsafe intrusion by other drivers. At signal controlled intersections, stop 10 ft. behind crosswalk or other vehicle. At stop sign controlled intersections, approach stop sign cautiously and ascertain if cross traffic has to stop. Stop at or just behind limit line or crosswalk.
12	Get the big picture.	Collision, injury or death to occupants or other parties.	Avoid being unnecessarily boxed in. Avoid sudden acceleration and deceleration. Maintain a minimum of 4 second following distance, adjust speed to traffic conditions, scan immediate and adjacent lanes before merging.

No	Job Steps	Potential Hazard(s)	Critical Action(s)
13	Make sure they see you.	Collision, injury or death to occupants or other parties.	Seek eye contact with other drivers or pedestrians. Cover or use horn when conditions warrant. Before changing lanes, signal well in advance, check mirrors and over shoulder, and allow adequate space before changing lanes. Break early to activate brake lights. Stay out of other vehicle blind spots. Gently sound horn or flash lights if unsure other driver or pedestrian sees you. Turn on headlamps in high traffic areas, at dusk, and in inclement weather.
14	Backing up.	Collision, injury or death to occupants or other parties.	Make all backing maneuvers slowly and cautiously. Check mirrors and over shoulders. When parking, look for pull-through parking space away from traffic to avoid backing out of a parking space.
15	Parking.	Collision, injury or death to occupants or other parties.	Park away from other cars and traffic. Back into parking spot when possible and safe. Use drive through spaces when available. Maintain cushion of safety from fixed objects. Set parking brake.
16	POST-TRIP - Report maintenance or mechanical problems upon returning vehicle.	Conditions worsen leading to mechanical failure resulting in accident, injury or death.	Report vehicle problems immediately to company representative or rental car agency.

No	Job Steps	Potential Hazard(s)	Critical Action(s)
	OFF ROAD DRIVING	Collision, injury or death to occupants or other parties.	<p>When driving off-road drive <i>as slow as possible and as fast as necessary</i>, avoid excess speed, drive on previously used paths, walk the path first to determine if you can negotiate mud, sand, or other obstacles, make sure your thumbs are safely on the outside of the steering wheel, know your vehicle clearance, use a spotter outside the vehicle for obstacles; engage 4-wheel drive before you need it.</p> <p>When riding through deep sand, snow or mud, deflate your tires slightly to increase the tire's footprint and provide better traction. Deflated tires will decrease your ground clearance though. Remember to re-inflate your tires before going on-road again.</p> <p>Use a steady momentum to carry you through. Keep your speed up and use higher gears. Don't spin your tires, and don't stop till you're out of the deep sand. If your wheels start to spin, ease off the throttle just a bit and allow the tires to slowdown and regain traction.</p> <p>If you lose traction and the vehicle is barely moving, turn the steering wheel quickly from side to side in short strokes (only 1/8th turn) to allow the front tire walls to find extra grip.</p> <p>If muddy conditions force you to drive in the ruts, know where your front wheels are pointed at all times. Your vehicle will follow the ruts, even with the wheels turned to the right or left. If you encounter a dry spot with the wheels turned, then the front wheels can regain traction and suddenly throw the vehicle out of the ruts, resulting in a loss of control and possible damage.</p>

<b>Job Hazard Analysis – Soil Sampling</b>	
Field staff must review job-specific work plan and coordinate with project manager to verify that all up-front logistics are completed prior to starting work including, but not limited to, permitting, access agreements and notification to required contacts (e.g., Site managers, inspectors, clients, subcontractors, etc.). Additionally, a tailgate safety meeting must be performed and documented at the beginning of each work day. Also consider weather conditions (heat, cold, rain, lightning).	
<b>Date:</b>	March 2013
<b>JHA Type:</b>	
<b>Work Type:</b>	Soil Sampling
<b>Work Site:</b>	DEPO
<b>Organization:</b>	ECM
<b>Personal Protective Equipment (PPE) needed</b>	

No	Job Steps	Potential Hazard(s)	Critical Action(s)
1	Review JHA and HASP	Site hazards	Review JHAs and HASP; identify Site safety representatives, review hospital route.
2	Load/ unload vehicle.	Lifting	Get help and/or use proper equipment to assist with heavy lifting; bend at knees (not at back); keep objects close to your body; do not twist; minimize movement of heavy objects..
3	Soil sampling	Biologic hazards; contaminated soil, heat/cold stress; slip, trip, fall.	Wear appropriate PPE including chemical resistant gloves; wash with soap and water after conducting sampling activities, do not eat or chew gum while conducting sampling activities; be cautious of footing, loose soil, or wet ground; select sampling time during temperate time of year (not the hottest or coldest); collect samples while stirring up a minimum amount of dust, sample areas that are easily accessed, and avoid areas being occupied by wildlife, bugs, insects, etc.
4	Use of hand tools	Pinch points, hand injuries	Ensure that the proper tool is identified and used for the job at hand; ensure the tool is used properly in accordance with its design.
5	Decontamination.	Contaminated materials.	Wear appropriate PPE including chemical resistant gloves; wash with soap and water after conducting sampling activities, do not eat or chew gum while conducting decontamination activities

No	Job Steps	Potential Hazard(s)	Critical Action(s)
6	Solid/liquid waste management.	Exposure to contaminated materials, lifting/moving containers; slip, trip, fall.	Wear appropriated PPE including chemical resistant gloves and eye protection; watch for pinch points when closing containers; be cautious when lifting or moving containers to avoid back or hand injury; get assistance in moving heavier containers; be cautious of moving liquid containers as contents may shift or 'slosh;' be cautious of footing, loose soil, or wet ground when moving containers – all these conditions can cause unstable conditions that may result in a slip, trip or fall.

<b>Job Hazard Analysis – Subcontractor Oversight</b>	
Field staff must review job-specific work plan and coordinate with project manager to verify that all up-front logistics are completed prior to starting work including, but not limited to, permitting, access agreements and notification to required contacts (e.g., Site managers, inspectors, clients, subcontractors, etc.). Additionally, a tailgate safety meeting must be performed and documented at the beginning of each work day. Also consider weather conditions (heat, cold, rain, lightning).	
<b>Date:</b>	March 2013
<b>JHA Type:</b>	
<b>Work Type:</b>	Job Hazard Analysis – Subcontractor Oversight
<b>Work Site:</b>	DEPO
<b>Organization:</b>	ECM
<b>Personal Protective Equipment (PPE) needed</b>	
Safety gloves (type dependent on job-specific requirements_	
Boots	
Safety glasses	
First Aid Kit	

No	Job Steps	Potential Hazard(s)	Critical Action(s)
1	Contractor Oversight	Personal Injury, equipment damage, lost time incidents, slips, trips, falls	Be aware of surroundings, follow safe work practices and procedures Equipment: Keep eye contact with equipment operators before entering work zones. Make sure equipment operators know you are in the area. Let equipment operators know when you leave the area. Traffic: Be mindful of oncoming traffic when entering/exiting work zone. Be sure to remain within traffic control zone situated with traffic barriers between you and oncoming traffic. Site conditions: Identify and mitigate trip, slip and fall hazards to the extent possible. If conditions cannot be mitigated, alert personnel. Make sure all walking paths are free of hazards. Weather: Apply sun screen as needed to exposed skin. Drink water frequently. Wear rain gear if necessary.
2	Documentation.	Poor records	Always document Site activities, equipment usage, sampling areas/descriptions, work conducted in accordance with acceptable Site procedures, Plans, and Specifications of the project. Always QA/QC field notes. Be sure to meet or exceed customer requirements.
3	Decontamination.	Migration of COCs Contact with COCs	Minimize potential for cross contamination. Dispose of nitrile gloves before leaving the exclusion zone or Site. Wash boots as appropriate. Consider bringing and extra pair of clothes/shoes to change into before driving personal vehicles.

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## **Appendix C: Exposure Monitoring for Thermal Stress**

## APPENDIX C: EXPOSURE MONITORING FOR THERMAL STRESS

### 1. HEAT STRESS

High ambient temperature can result in health effects ranging from transient heat fatigue, physical discomfort, reduced efficiency, personal illness, increased accident probability, etc., to serious illness or death. Heat stress is of particular concern when chemical protective garments are worn. It is important to keep in mind that protective clothing limits the dissipation of body heat and moisture, causing discomfort, inefficiency, and impaired functional ability. Wearing personal protective equipment places employees at considerable risk of developing heat stress. Under these circumstances, the probability of an accident occurring increases.

Heat stress is caused by a number of interacting factors, including environmental conditions, clothing, workload, and the individual characteristics of the worker. Because heat stress is probably one of the most common (and potentially serious) illnesses, regular monitoring and other preventive precautions are vital. The types of heat stress typically encountered during field activities and the associated first-aid conditions appear in sections 6.2.1 to 6.2.4 below.

The initiation of heat stress monitoring will be required when employees are working in environments exceeding 90°F ambient air temperature. If employees are wearing impermeable clothing, this monitoring will begin at 78°F. If workers exhibit heat stress symptoms, the heart rate and body temperature will require monitoring during all tasks (as the SHSO deems necessary or appropriate). It is anticipated that this monitoring can be self-performed once the health and safety representative demonstrates appropriate techniques to affected employees. Since individuals vary in their susceptibility to heat, this type of monitoring has its advantages. The two parameters that are to be monitored at the beginning of each rest period are heart rate and temperature. The action guidelines (USEPA, 1992) are as follows:

Heart rate: Count the radial pulse during a 30-second period as early as possible when at rest. If the heart rate exceeds 110 beats per minute at the beginning of the rest period, shorten the next work cycle by one-third and keep the rest period the same. If the heart rate still exceeds 110 beats per minute at the next rest period, shorten the following work cycle by one-third.

Body Temperature: Determine body temperature at the end of the work cycle and before drinking fluids. If the temperature is greater than 99.6°F, shorten the next work cycle by one-third without changing the rest cycle. Repeat. Do not permit workers to wear semi-permeable or impermeable clothing when their body temperature exceeds 100.6° F.

The SHSO shall log all heart-rate monitoring data in the field logbook. One or more of the following courses of action will reduce the probability of stress from climatic conditions, particularly from heat stress:

- Provide adequate break periods, including refreshments (hot drinks in cold weather and cold drinks in hot weather) for the type of work being conducted.
- Establish a work schedule that will provide sufficient rotation of team members in and out of stressful situations/tasks.
- Wear sufficient cooling devices under protective clothing, but use caution, as these layers add bulk, decrease mobility, and contribute to fatigue.
- Use portable showers and hose-downs in extremely hot situations.
- Use cooling vests.
- Provide areas of shade on-Site if possible or employ use of picnic umbrellas.
- Schedule work to avoid the hottest part of the day.



Note that these guidelines assume that a worker is acclimated to ambient temperature on Site. This physiological adjustment can generally take several days to occur. In order that a new worker on-Site has an opportunity to safely acclimate, ECM will adjust the suggested work/rest regimes to a level where the worker does not feel unduly heat stressed the first several days of work. It is also important that work/rest cycles match the exertion level of the particular Site activities (e.g., standing observer versus active sampler).

## **1.1 HEAT RASH**

Heat rash can be caused by continuous exposure to hot and humid air and skin abrasion from sweat soaked clothing.

- **Signs and Symptoms:** The condition is characterized by a localized red skin rash and reduced sweating. Aside from being a nuisance, the ability to tolerate heat is reduced.
- **Treatment:** Keep skin hygienically clean and allow it to dry thoroughly after using chemical protective clothing.

## **1.2 HEAT CRAMPS**

Heat cramps result from heavy exertion and profuse sweating with inadequate electrolyte fluid replacement. This often robs the larger muscle groups (stomach and legs) of blood, which can cause painful muscle spasms and pain.

- **Signs and Symptoms:** Muscle spasms and pain in the extremities and abdomen.
- **Treatment:** Remove employee to a cool place protected from direct sunlight and give sips of water or an electrolytic drink. Apply manual pressure to the cramped muscle. Watch for signs of heat exhaustion or stroke.
- Transport the individual to a hospital if the condition worsens or if there is any indication of a more serious problem.

## **1.3 HEAT EXHAUSTION**

Heat exhaustion is a mild form of shock caused by increased stress on various organs to meet increased demand to cool the body. Sweat may not evaporate properly due to high humidity or layers of clothing, resulting in inadequate cooling of the body. Onset is gradual and symptoms should subside within one hour if properly treated.

- **Signs and Symptoms:** Weak pulse; shallow breathing; pale, cool, moist skin; profuse sweating; dizziness; fatigue.
- **Treatment:** Remove employee to a cool place and remove as much clothing as possible. Give sips of water or electrolytic solution and fan the person continually to remove heat by convection. Allow the person to lie down and elevate the feet above ground. CAUTION: Do not allow the affected person to become chilled—treat for shock if necessary.
- Transport the individual to a medical facility if victim's condition worsens or if there is any indication of a more serious problem.

## **1.4 HEAT STROKE**

Heat stroke is the most severe form of heat stress; the body must be cooled immediately to prevent severe injury and/or death. THIS IS A MEDICAL EMERGENCY!!

- Signs and Symptoms: Red, hot, dry skin; body temperature of 105 degrees Fahrenheit (EF) or higher; no perspiration; nausea; dizziness and confusion; and strong, rapid pulse.
- Treatment: Heat stroke is a true medical emergency. Transportation of the victim to a medical facility must not be delayed. Prior to transport, remove as much clothing as possible and wrap the victim in a sheet soaked with water. Fan the victim vigorously while transporting to help reduce body temperature. Apply cold packs, if available; place under the arms, around the neck, or any other place where they can cool large surface blood vessels. If transportation to a medical facility is delayed, reduce body temperature by immersing victim in a cool water bath (however, be careful not to over-chill the victim once body temperature is reduced below 102° F). If this is not possible, keep victim wrapped in a sheet and continuously douse with water and fan.

## 1.5 PREVENTION

The implementation of preventative measures is the most effective way to limit the effects of heat-related illnesses. During periods of high heat, adequate liquids must be provided to replace lost body fluids. Replacement fluids can be a first aid electrolyte replacement solution, a commercial mix such as Gatorade, or a combination of these with fresh water. The replacement fluid temperature should be kept cool, 50° F to 60° F, and should be placed close to the work area. Employees must be encouraged to drink more than the amount required to satisfy thirst. Employees should also be encouraged to salt their foods more heavily during hot times of the year. In high heat personnel should stay out of direct sun when possible. Also, work may be scheduled to avoid the hottest times of the day.

**Sunburns** are another hazard of performing outdoor work. If hard hats are not necessary, team members should consider a brimmed hat and possibly neck flaps. Many weather reports now include an ultraviolet index to aid in the determination to apply sunscreen. When using sunscreen it is important to get one with a sun protection factor of about 30. Apply the sunscreen at least 30 minutes prior to going outdoors and reapply during the day.

Cooling devices such as vortex tubes or cooling vests can be worn beneath impermeable clothing. If cooling devices are worn, only physiological monitoring will be used to determine work activity. All workers are to rest when any symptoms of heat stress are noticed. Rest breaks are to be taken in a cool, shaded rest area. Employees shall remove chemical protective garments during rest periods and will not be assigned other tasks.

All employees shall be informed of the importance of adequate rest and proper diet in the prevention of heat stress and the harmful effects of excessive alcohol and caffeine consumption.

## 2. COLD STRESS

On days with low temperatures, high winds, and humidity, anyone can suffer from the extreme cold. Severe cold exposure can be life threatening. Several factors increase the harmful effects of cold: being very young or very old, wet clothing, having wounds or fractures, smoking, drinking alcoholic beverages, fatigue, emotional stress, and certain diseases and medications.

Cold weather injuries may be local or systemic. Local cold weather injuries include chilblains (chronic injury of the skin and peripheral capillary circulation) and frostbite. Frostbite occurs in three progressive stages: frostnip, superficial frostbite, and deep frostbite. Systemic cold injuries, due to hypothermia, are those that affect the entire body system. Hypothermia is caused by exposure to cold and is aggravated by moisture, cold winds, fatigue, hunger and inadequate clothing or shelter. Precautionary measures that will be taken include:

- Providing field shelters or wind screens
- Monitoring temperature and wind speed to determine appropriate cold stress personal safety measures
- Adjusting work schedule based on weather conditions and temperature
- Providing insulated clothing for field workers
- Adhering strictly to the buddy system so that workers can assess cold stress symptoms in their co-workers.

**Frostbite Monitoring.** Frostbite is a potentially crippling condition that can occur when inadequately protected skin or body parts are subjected to freezing weather. All team members should continually be alert for signs of frostbite in coworkers and bring it to the attention of the SHSO. A cold feeling, pain, and numbness precede the onset of frostbite. Frostbite usually appears as gray or white waxy spots on skin. Areas most susceptible are nose, ears, and cheeks.

The following steps should be taken to avoid frostbite:

- Dress warmly (avoid cotton, wear polypropylene, wool, gortex or other moisture wicking materials instead)
- Wear layers of clothes
- Keep boots and gloves loose-fitting
- Stay dry; carry extra clothing
- Avoid touching cold metal with bare hands
- Avoid spilling cold fuel, alcohol, or other liquids that freeze below 32°F on your body or clothing.

If a person suffers frostbite, get them to a hospital as soon as possible. If transport to a hospital is not immediately available, get the person to a warm shelter and immediately perform the following:

- Cover exposed areas with additional clothing while still exposed to the elements.
- Wrap the person in blankets or a sleeping bag.
- Give the person warm drinks (no liquor).
- Undress the frozen part and submerge the frozen part in a tub of warm water (102°F to 105°F), or put the frostbitten person in a large tub of warm water, if available, and stir the water.
- Warm with skin to skin contact, such as placing warm hands on frozen nose or ears, but do not rub.
- Get the person to a hospital as soon as possible.

Do **not** allow the following to occur:

- Do not rub the frozen part.
- Do not give the person liquor.
- Do not allow the person to walk on thawed feet.
- Do not let the person smoke.

- Do not break any blisters that may form.
- Do not let the thawed part freeze again.
- Do not warm the frozen part in front of a source of dry heat (open fire, oven, *etc.*).

**Hypothermia Monitoring.** Hypothermia is a lowering of the body's temperature due to exposure to cold or cool temperatures. All team members should continually be alert for signs of hypothermia in co-workers and bring it to the attention of the SHSO. Most cases of hypothermia occur at temperatures between 30°F and 50°F. If not properly treated, hypothermia can cause death. Safety equipment for hypothermia should include a synthetic sleeping bag and a hypothermia thermometer. ***HYPOTHERMIA IS A MEDICAL EMERGENCY!*** Transport to a hospital as soon as possible, even if victim appears to be recovering.

To prevent hypothermia:

- Eat well prior to exposure.
- Dress warmly (avoid cotton, wear polypropylene, wool, gortex or other moisture wicking materials instead).
- Avoid becoming wet due to sweating, rain or snow, or falling in water.

Early signs of hypothermia may include:

- Violent shivering
- Slurred speech
- Decrease in coordination
- Confusion, inability to answer simple questions
- Unusually irritable behavior
- Strange behavior
- Tendency to drop or lose clothing or equipment

As hypothermia progresses into more serious stages victims typically:

- Develop trouble seeing clearly
- Become sleepy and numb
- Move with difficulty
- Eventually become unconscious, if not properly cared for.

The following actions should be taken to treat a hypothermia victim:

- Get the victim to a warm, dry shelter as soon as possible.
- Remove any wet or cold garments and dry the person thoroughly.
- Wrap the victim in blankets, sleeping bags or dry clothing to prevent more heat loss.
- If a warm area is not available:
  - Build a shelter and put the victim in the warmest, driest area available.

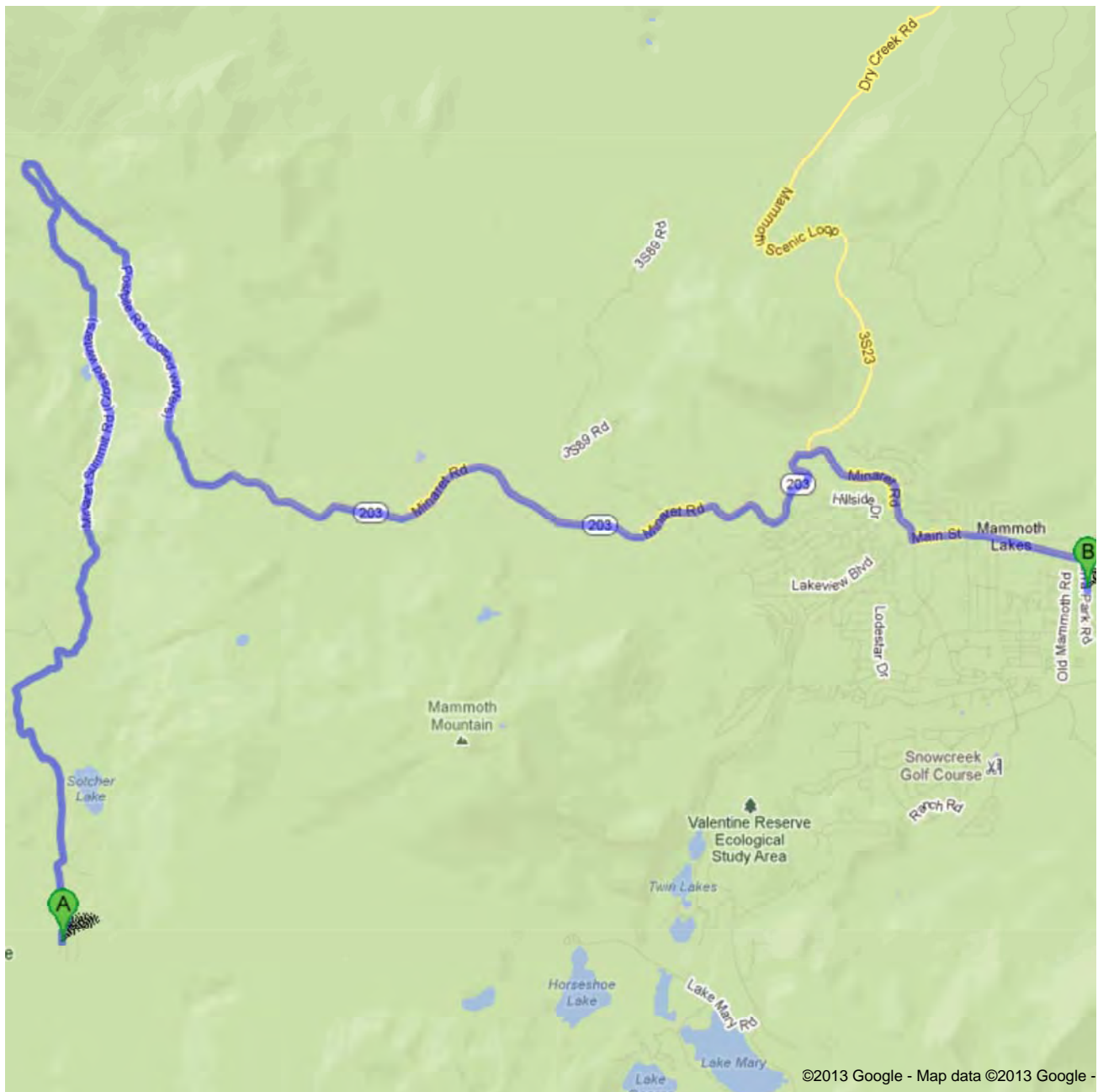
- Remove any wet or cold garments.
  - Have one or more persons remove their clothing and lay next to the victim, providing skin to skin contact.
  - Wrap the victim and rescuers in dry warm blankets, sleeping bags or clothing.
  - When the victim becomes conscious, place warm objects along the victim's sides to warm vital areas.
- When the victim is able to swallow easily, provide warm, sweetened drinks and food (preferably candy or sweetened food).
- Do not give the victim alcohol or allow to smoke.
- Do not rub the victim's skin.
- Keep checking the victim and give additional assistance as needed.

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## **Appendix D: Hospital Route Maps**



**Directions to Mammoth Hospital**  
85 Sierra Park Rd, Mammoth Lakes, CA 93546  
**14.9 mi – about 35 mins**



**Devils Postpile National Monument**

Madera, CA

- |   |              |
|---|--------------|
| 1. Head <b>north</b> on <b>Reds Cir</b>   | go 0.1 mi    |
| <b>This road may be seasonally closed</b> | total 0.1 mi |



- |  |              |
|--|--------------|
| 2. Turn left toward <b>Minaret Summit Rd</b> | go 0.7 mi    |
| <b>This road may be seasonally closed</b>    | total 0.8 mi |
| About 2 mins                                 |              |

- |  |              |
|--|--------------|
| 3. Continue straight onto <b>Minaret Summit Rd</b> | go 1.9 mi    |
| <b>This road may be seasonally closed</b>          | total 2.8 mi |
| About 5 mins                                       |              |

- |   |              |
|---|--------------|
| 4. Continue onto <b>Reds Creek</b>        | go 0.3 mi    |
| <b>This road may be seasonally closed</b> | total 3.1 mi |

- |   |              |
|---|--------------|
| 5. Continue onto <b>Minaret Summit Rd</b> | go 2.5 mi    |
| <b>This road may be seasonally closed</b> | total 5.5 mi |
| About 6 mins                              |              |



- |   |              |
|---|--------------|
| 6. Slight right onto <b>3511 Forest Rd/Lookout Point Rd/Postpile Rd</b> | go 2.7 mi    |
| <b>This road may be seasonally closed</b>                               | total 8.2 mi |
| About 7 mins  |              |



- |  |               |
|--|---------------|
| 7. <b>3511 Forest Rd/Lookout Point Rd/Postpile Rd</b> turns slightly right and becomes <b>Minaret Rd</b> | go 5.4 mi     |
| <b>This road may be seasonally closed</b>  | total 13.6 mi |
| About 11 mins  |               |



- |                                  |               |
|----------------------------------|---------------|
| 8. Turn left onto <b>Main St</b> | go 1.1 mi     |
| About 3 mins                     | total 14.7 mi |



- |  |               |
|--|---------------|
| 9. Turn right onto <b>Sierra Park Rd</b> | go 0.2 mi     |
| Destination will be on the left          | total 14.9 mi |

**Mammoth Hospital**

85 Sierra Park Rd, Mammoth Lakes, CA 93546

These directions are for planning purposes only. You may find that construction projects, traffic, weather, or other events may cause conditions to differ from the map results, and you should plan your route accordingly. You must obey all signs or notices regarding your route.

Map data ©2013 Google

Directions weren't right? Please find your route on [maps.google.com](http://maps.google.com) and click "Report a problem" at the bottom left.