



Tulsa Port of Inola Wasteload Allocation Work Plan



Tulsa Ports

Tulsa Port of Inola Industrial Park Project No. 134600

> Revision 1 February 24, 2022



Tulsa Port of Inola Wasteload Allocation Work Plan

prepared for

Tulsa Ports Tulsa Port of Inola Industrial Park Inola, Oklahoma

Project No. 134600

Revision 1 February 24, 2022

prepared by

Burns & McDonnell Engineering Company, Inc. Oklahoma City, Oklahoma,

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LIST OF ABBREVIATIONS

Abbreviation	Term/Phrase/Name
7Q2	Seven-Day, Two-Year Low Flow
Burns & McDonnell	Burns & McDonnell Engineering Company, Inc.
BOD20	Biochemical Oxygen Demand, 20-Day
City	City of Inola
CBOD ₅	Carbonaceous Biochemical Oxygen Demand, 5-day
CBOD ₂₀	Carbonaceous Biochemical Oxygen Demand, 20-day
CTOO2	USACE Verdigris River near Catoosa, OK Stream Gauge
DO	Dissolved Oxygen
EPA	United States Environmental Protection Agency
Ka	Reaeration Coefficient
INLO2	USACE Newt Graham Lock and Dam 18 Stream Gauge
MGD	Million Gallons per Day
mg/L	Milligrams per Liter
mL	Milliliter
MoS	Margin of Safety
NOAA	National Oceanic and Atmospheric Administration
NPDES	National Pollutant Discharge Elimination System
NPS	Non-Point Source
ODEQ	Oklahoma Department of Environmental Quality
Port of Inola	Tulsa Port of Inola Industrial Park
POTW	Publicly Owned Treatment Works

Abbreviation	Term/Phrase/Name
Q	Volumetric Flow Rate
QAM	Quality Assurance Manual
RMSE	Root Mean Square Error
S	Channel Slope
U	Mean Water Velocity
USACE	United States Army Corps of Engineers
WLA	Waste Load Allocation
WQS	Water Quality Standards
WWAC	Warm Water Aquatic Community
WWTP	Wastewater Treatment Plant

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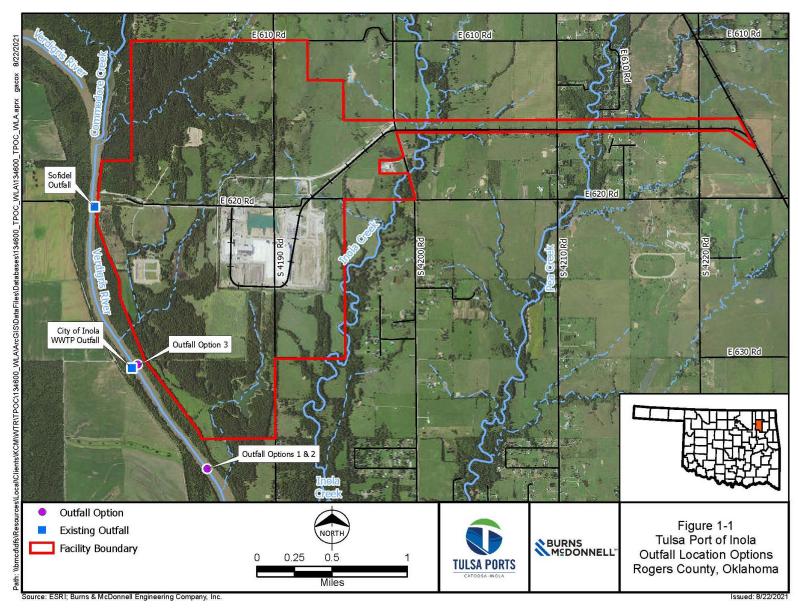
1.0 **PROJECT OVERVIEW**

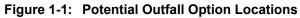
The purpose of this project is to perform a Waste Load Allocation (WLA) study for the Tulsa Port of Inola Industrial Park (Port of Inola). Burns & McDonnell Engineering Company Inc. (Burns & McDonnell) has been retained by the City of Tulsa-Rogers County Port Authority (Tulsa Ports) to develop the WLA for this site. Burns & McDonnell has subcontracted the field sampling and lab tests to Green Country Testing and subcontracted the QUAL2K modeling to Carollo Engineers.

1.1 Purpose and Background

The purpose of this document is to provide a work plan for the field surveying/sampling in preparation of a WLA study as part of the Port of Inola Industrial Park. The proposed development will consist of the construction of an industrial park that will consist of a variety of industries that encompass manufacturing, industrial, and warehousing. The industrial park site is comprised of approximately 2,400 acres of undeveloped land located southwest of the intersection of E 620 Rd and S 4200 Rr and adjacent to the Verdigris River in the City of Inola (City), Oklahoma's corporate boundary. A wastewater treatment plant is proposed that will treat both the industrial and domestic discharges from the industrial park. Additionally, the Port of Inola may acquire the City's domestic wastewater and the associated waste load allocations in NPDES permit OK0033618. Figure 1-1 shows the three discharge scenarios that will be assessed.

1-1





1.1.1 Discharge Option 1

Option 1 will evaluate the discharge of only domestic and industrial wastewaters generated onsite by the Port customers. The wastewaters would be piped approximately two (2) miles south of the Port and discharged to the Verdigris River (36.094401°, -95.555803°), immediately downstream of the City of Inola's mixing zone.

The Port is currently in the process of soliciting customers for the industrial park. Therefore, the exact composition of industrial wastewaters cannot be identified at this time. However, the following list of target industries serves as a general guide for the types of industrial wastewaters that will be produced on site:

- Plastic and resin manufacturing
- Nonferrous metal rolling and alloying
- Iron and steel manufacturing
- Metal stamping and forging
- Structural metal product manufacturing
- Engine and turbine manufacturing
- Car and automobile manufacturing
- Aircraft, engine, and parts manufacturing

Based on current interest, the Port is expecting approximately 0.3 million gallons per day (MGD) of domestic wastewater and 2.0 MGD of industrial wastewaters for the initial phase of the industrial park, and 0.7 MGD and 3.3 MGD of domestic and industrial, respectively, wastewater (total) to be produced once the industrial park is fully built out. This would result in a total design discharge of 2.3 MGD for the initial phase of the industrial park and 4.0 MGD for the final phase of the industrial park. Table 1-1 summarizes the flows for these phases.

Flow Component	Initial Phase Flows (MGD)	Final Phase Flows (MGD)
Industrial Wastewater	2.0	3.3
Domestic Wastewater	0.3	0.7
Industrial Area Employees	0.3 (9,400 employees*)	0.7 (18,800 employees*)
City of Inola	0	0
Total Wastewater Projection	2.3	4.0

Table 1-1: Discharge Option 1 Flow Projections

*It was assumed that industries would operate with two to three 8-hour shifts. Flows were estimated based on 15 gallons/person/day (Metcalf and Eddy, 2014) and 2.2 shifts on average; two day shifts that operate at full capacity, and one night shift that operates at 20% capacity.

1.1.2 Discharge Option 2

In addition to the wastewaters produced by the facilities onsite of the industrial park discussed in Option 1, the Port of Inola is also considering acquiring, treating, and discharging the domestic wastewater for the City's Wastewater Treatment Plant (WWTP), NPDES permit OK0033618, which has a design flow of 0.4 MGD. The Port proposes to acquire the WLA that is currently included in the City's WWTP NPDES permit OK0022518 in addition to the available loadings identified in the WLA study. The City's WWTP currently operates as a Publicly Owned Treatment Works (POTW) but does not treat wastewater from industrial dischargers.

Burns & McDonnell determined the 20-year population projection using openly sourced data from the EPA's ECHO database and U.S. Census Bureau. Between 2000 to 2020, the City's population grew from 1589 to 1797 resulting in a 13% population growth. The 20-year population projection was determined by using the more conservative of either a 13% increase seen over the next 20-years or a 1.5% year over year (YOY) population increase. Based on the Discharge Monitoring Report (DMR) data from EPA's ECHO database, between 2019 to 2020 the City's WWTP discharged an average monthly average of 0.351 MGD and a max daily max of 0.498 MGD. The City's current population is 1,797 which results in a daily average of 195 gallons per capita per day (gpcd) and a daily max of 277 gpcd. These daily discharge numbers were then applied to the 20-yr population projections and the most conservative was used to determine the discharge volume for acquiring the City's discharges. Table 1-2 shows this comparison and the selected discharges (bolded) for this study.

		Discharge		
Percent Increase	20-Year Population Projection	Monthly Average (MGD)		
13% Increase	2031	0.396	0.563	
1.5% YOY	2040	0.472	0.670	

Table 1-2: City of Inola 20-Year Population Projection

The Port of Inola's combined wastewater flows for the initial discharge would be 2.7 MGD and based on the 20-yr population projections for the City of Inola and the Industrial Park operating at full capacity, the final design flow from Port of Inola would be 4.5 MGD. The discharge location for this option would be the same as Option 1 (36.094401°, -95.555803°). Table 1-3 itemizes these flow projections for each phase.

Flow Component	Initial Phase Flows (MGD)	Final Phase Flows (MGD)
Industrial Wastewater	2.0	3.3
Domestic Wastewater	0.7	1.2
Industrial Area Employees	0.3 (9,400 employees)	0.7 (18,800 employees)
City of Inola	0.4	0.5
Total Wastewater Projection	2.7	4.5

Table 1-3: Discharge Option 2 Flow Projections

1.1.3 Discharge Option 3

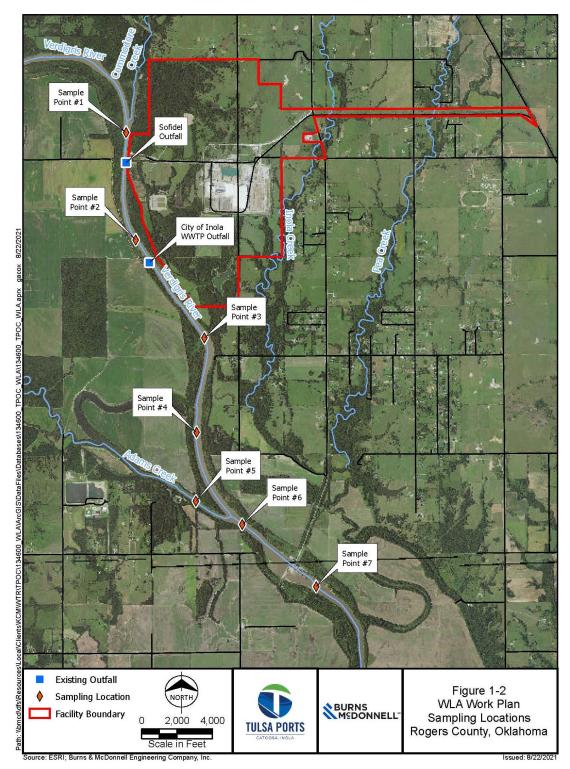
The third discharge option is the combination of the Port's wastewaters and the City's domestic wastewaters, discussed in Option 2, to be discharge to the Verdigris River via the City's current WWTP outfall located (36.103686°, -95.564694°). The Port of Inola's combined wastewater flows for the initial discharge would be 2.7 MGD and the final design flow from Port of Inola would be 4.5 MGD. Table 1-3 details these flow projections for the two phases.

1.2 Receiving Waters

The minimum dissolved oxygen (DO) concentrations are dependent upon the designated use for the river as specified in the Oklahoma Water Quality Standards (Oklahoma Administrative Code Title 785, Chapter 45) as published and updated by OWRB. Per the current Water Quality Standards, this reach of the Verdigris River, identified as stream segment 121500020120, is designated as a Warm Water Aquatic Community (WWAC), which governs the minimum allowable concentrations of DO during the spring (April 1st through June 15th) at 6.0 mg/L and 5.0 mg/L the rest of the year. To meet this criterion, less than 10 percent of the samples can fall below these levels for both time periods.

To evaluate that the minimum DO concentrations are maintained during these time periods, water quality modeling will be performed. The goal of the modeling is to simulate the Verdigris River during the critical periods of the year (i.e., high temperature, low flow) for DO concentrations. A new QUAL2K model will be developed and calibrated based on field data collected by the project team. The laboratory will collect samples from seven locations along approximately 30,000 feet of the Verdigris River for one sampling period. At each location, four replicate samples will be collected, totaling twenty-eight samples for analyses, see Figure 1-2.

The WLA will be determined from the model. WLA limits will then be generated from the WLA after a Margin of Safety (MoS) has been applied to the WLA. The MoS values are established by the Oklahoma Department of Environmental Quality (ODEQ) and listed in Table 1-4 from the ODEQ 2012 Continuing



Planning Process. As discussed previously with ODEQ, for additional conservatism and margin, a 25% MoS will be added to thresholds determined in this study for the WLA limit.

Figure 1-2: Sampling Locations

Model	System Complexity	Margin of Safety
Uncalibrated	Multiple Source/Complex Waste	25%
Uncalibrated	Single Source/Complex Waste	20%
Calibrated	Multiple Source/Complex Waste	15%
Calibrated	Single Source/Complex Waste	10%
Validated		5%

Table 1-4: ODEQ Margin of Safety Percentages based on Model Type and System Complexity

1.3 **Project Schedule**

Table 1-5 provides the projected dates of completion for the major project tasks. These dates are subject to change based on ODEQ comments and weather conditions for sampling.

Task	Start Date	End Date
Prepare Draft WLA Study Work Plan	July 2021	December 2021
ODEQ Review of Draft WLA Study Work Plan	December 2021	February 2022
Conduct Field Sampling	August 2021	August 2021
Receive Lab Analysis of Samples	September 2021	September 2021
Prepare Water Quality Model and Report	December 2021	March 2022
ODEQ Review of Model and Report	March 2022	April 2022
Finalize Model and Report	April 2022	April 2022

Table 1-5: Project Schedule

1.4 Data Collection

To establish model parameters, grab samples were collected on August 31, 2021 and September 1, 2021 at the seven locations depicted on Figure 1-2. A QUAL2K water quality model will be developed and calibrated using the grab sample data as well as other previous studies and publicly available information. Previous studies will be used to provide initial calibration parameters. However, these parameters will be adjusted during the calibration based on matching the model outputs to the grab sample data. Publicly available meteorological data from the NOAA Claremore Regional Airport weather station, Figure 1-3. Upstream flow data from the United States Army Corps of Engineers (USACE) CTOO2 Verdigris River

near Catoosa, OK (CTOO2) stream gauge and downstream flow data from the USACE Newt Graham Lock and Dam 18 (INLO2) stream gauge. The CTOO2 and Lock and Dam 18 gauges have data regarding stage level and flow rate.

The geographical extent of the sampling locations, Figure 1-2, will be the same as the geographical extent of the model. The sampling locations include six sampling points along the Verdigris River that start approximately 1,500 feet upstream of the Sofidel outfall and 11,000 feet upstream of the City's WWTP outfall (Discharge Option 3) to approximately 22,000 feet downstream of the Discharge Options 1 and 2 location. A sampling location has also been included on Adams Creek approximately 2,000 ft upstream of where Adams Creek enters the Verdigris River.

The samples were collected on August 31, 2021, which had a high of 93 °F with a 2% chance of precipitation. These conditions resemble the critical summertime conditions for high water temperature and low flow that is likely to occur in one out of every two years (7T2 and 7Q2, respectively), which is preferred for calibrating the water quality model.

For oxygen-demanding parameters, Oklahoma WQS define the seven-day, two-year low flow (7Q2) as the receiving stream flow for determining allowable discharge load to a stream. The flow is calculated as a moving average of seven consecutive days for each year or season in a given record and represents a yearly or seasonal low flow value. Seasonal 7Q2 flows for the Verdigris River will be calculated using flow data obtained from the USACE CTOO2 stream gauge located approximately 10 miles northwest from the project site. The most recent ten years of daily flow measurements will be used to calculate a 7Q2. The USACE INLO2, Newt Graham Lock and Dam 18 (INLO2) stream gauge was also reviewed for flow data, but due to operations at the Newt Graham Lock and Dam, the INLO2 stream gage flow data contained uncharacteristically low flows for certain periods of the year along with artifacts in the data that questions the validity of the dataset, particularly for low flow conditions. Outside of the low flow days, the flows between the CTOO2 and INLO2 stream gauges mirrored each other, see Figure 1-4. In addition, daily flow data for the nearest upstream USGS stream gages (Verdigris River near Claremore and Bird Creek at State Highway 266 near Catoosa) were used to verify the flow data for USACE CTOO2. Therefore, it was determined that flow data from the CTOO2 stream gauge would be used in developing the seasonal 7Q2 flow rates used in the QUAL2K model. Given the highly controlled hydraulics in this reach, and the lack of minor streams and tributaries entering the Verdigris River, the flow measured at the CTOO2 stream gauge is equivalent to the flow at the proposed Port of Inola discharge point. Flow will be provided by the upstream CTOO2 station. Cross-sections will be assumed to be consistent throughout the model domain (i.e., constant channel depth and width).

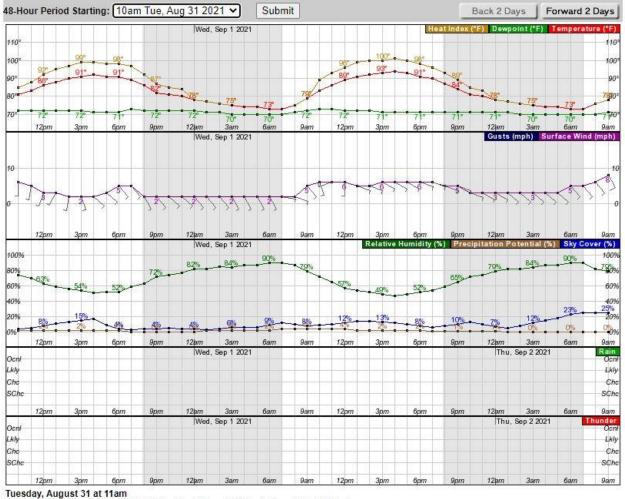
A base assumption in the modeling/calibration scope is that the geometry of the Verdigris River is relatively static between Catoosa and Lock and Dam 18. The Verdigris River is regularly dredged and maintained by the USACE; therefore, the channel depth and width will be assumed constant throughout the model domain. To estimate a channel slope that would represent the required 9-ft navigable depth throughout the modeled river segment and represent the dam and lock operation, it was assumed that the water surface elevation (WSE) at Lock and Dam 18 would remain constant regardless of flow, thus the weir flow would be equal to the river flow. Because WSE in the river at Lock and Dam 18 would be relatively constant it was assumed that the WSE recorded by Digital Elevation Maps (DEM) and available from the United States Geological Survey (USGS) would be representative of typical WSE and could be used in conjunction with the navigable depth assumption to estimate an upstream water depth. Assuming that the channel depth on the upstream side of Lock and Dam 18 in the Sofidel model is representative of actual conditions and a constant bed slope between the headwater and Lock and Dam 18, a linear relationship can be used to determine the depth of the other model reach segments. Knowing the channel width, channel depth, manning's n, and flow rate, the slope of the hydraulic grade line using the Continuity of Flow and Manning's equations can be estimated. The calculated slope for each model reach upstream of Lock and Dam 18 was input as the slope for the manning formula in Qual2K.

The Oklahoma WQS require that allowable loadings to meet dissolved oxygen criteria be calculated using the seven-day, two-year low flow and the appropriate seasonal temperature. The values for the appropriate seasonal temperature are given in the Oklahoma WQS as a seasonal temperature associated with a particular fishery class, applicable season date, and associated DO criteria. Applicable temperatures for WWAC from Table 1 of OAC 785:45 Appendix G are summarized in Table 1-6.

WWAC	Dates Applicable	Seasonal Temperature (°C)
Early Life Stages	4/1 - 6/15	25
Other Life Stages		
Summer Conditions	6/16 - 10/15	32
Winter Conditions	10/16 - 3/31	18

 Table 1-6:
 Seasonal Temperatures for WWAC

Weather Elements	Fire Weather	Probabilistic Forecasts (Experimental) Description Survey
 Temperature (°F) Dewpoint (°F) Heat Index (°F) Surface Wind mph Sky Cover (%) Precipitation Potential (%) Relative Humidity (%) 	Mixing Height x100ft V Haines Index Trans. Wind mph V 20ft Wind mph V Vent Rate (x1000 mph-ft)	Quantitative Precipitation 6-hr 🖌 info
Rain Thunder Fog		



Temperature: 83 °F Dewpoint: 72 °F Heat Index: 88 °F Surface Wind: S 5mph Sky Cover (%): 5% Precipitation Potential (%): 2% Relative Humidity (%): 70% Rain: <10% Thunder: <10%



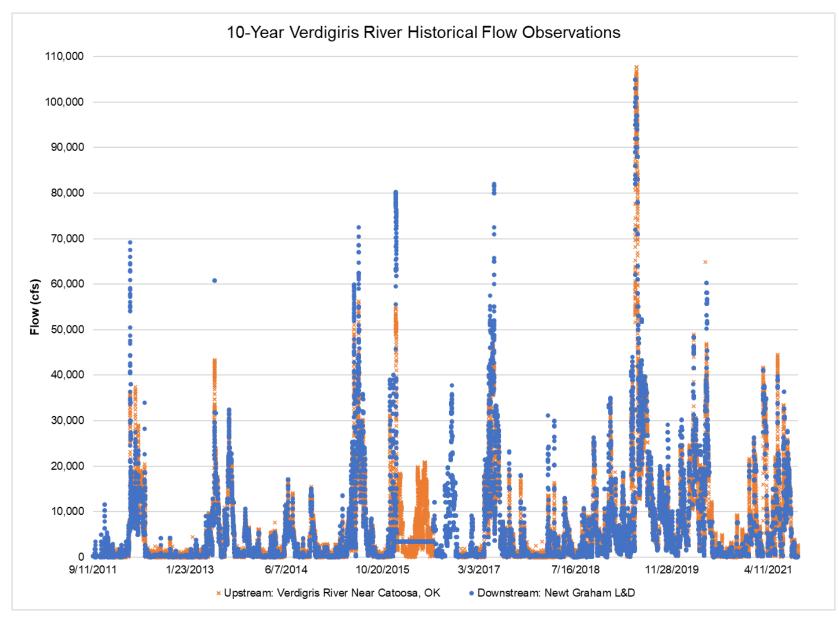


Figure 1-4: Flow Data USACE CT002 vs. INLO2 (2011 to 2021)

1.4.1 Model Development and Calibration

The QUAL2K model (Chapra et al., 2012) requires information about the local hydraulics, water quality and meteorology. This information is important to develop and calibrate the model by comparing it to measured values. For this model, various physical parameters are to be obtained from a site-specific field exploration:

The water grab samples will be analyzed by Green Country Testing for the following parameters:

- CBOD₅ (Filtered)
- CBOD₅ (Unfiltered)
- CBOD₂₀ (Filtered)
- CBOD₂₀ (Unfiltered)
- BOD₂₀
- Total Kjeldahl Nitrogen TKN
- Ammonia Nitrogen NH₃
- Nitrate $-NO_3$
- Nitrite $-NO_2$
- Total Phosphorus
- Orthophosphorus
- Enterococci
- E. coli
- Chlorophyll-a
- Total Suspended solids
- Volatile Suspended Solids
- Total Organic Carbon
- Dissolved Organic Carbon
- Alkalinity

The reaeration coefficient (Ka) will be determined by using the empirical Tsivoglou-Neal formulae

(Chapra et al., 2012):

- Ka = 31,183 US for low flow (Q < 15 cfs)
- Ka = 15,308 US for high flow (Q > 15 cfs)
- Ka = reaeration coefficient (units per day);
- U = mean water velocity (m/s) and
- S = channel slope (m/m).

In addition, Green County Testing collected data characterizing the pH, dissolved oxygen (DO), temperature, and specific conductivity of the water during the sampling collection. However, continuous *in situ* measurements will not be collected.

The model will be calibrated to the day of data collection, a period of high temperatures and low flow, which corresponds to higher nutrient concentrations and lower dissolved oxygen concentrations. The calibration parameters include all the environmental factors listed above. Root Mean Square Error (RMSE) and other traditional calibration scalars will be used to set the model. No validation of the model will be performed as there is no scheduled second field sampling event.

QUAL2K requires meteorological data (e.g., air temperature, dew point temperature, wind speed). Burns & McDonnell proposes to use the National Oceanic and Atmospheric Administration (NOAA) meteorological station at the Claremore Regional Airport (Figure 1-3), approximately 11 miles north-northeast of the project site for the required meteorological data input.

The model will be simulated for each of the three discharge options. The number of point sources and associated wastewater volumes will be modified to match each discharge option. Each discharge option will include the Sofidel discharge, which is located approximately 1,500 ft downstream of Sample Point 1. The City's discharge, located approximately 1,500 ft downstream of Sample Point 2, would be included in Discharge Options 1 (City only wastewater) and Discharge Option 3 (combined City and Port of Inola wastewaters). The third possible point source location would be located approximately 800 ft upstream of Sample Point 3. This location would be included for Discharge Option 1 (Port of Inola only wastewaters) and Discharge Option 2 (combined City and Port of Inola wastewaters). No non-point sources (NPSs) will be accounted in the model domain.

1.4.2 **Project and Sensitivity Simulations**

After the model is properly calibrated, sensitivity simulations will be performed with critical conditions (high temperatures, low flows) with the Port of Inola design discharge. The purpose of these simulations is to investigate different effluent parameters to determine point source limits that will not cause violations of DO standards. Included in the recommended WLA limits will be 25 percent margin of safety, which is conservative for the range typically applied to calibrated models (10-15 percent).

1.4.3 Report

After the model has been calibrated for water quality constituents and the additional simulations complete, a report will be originated and submitted to ODEQ to summarize the work performed and the

results of the WLA Study. This report will be used to establish the effluent requirements for the Port of Inola discharge into Verdigris River.

2.0 SAMPLING ACTIVITIES

The purpose of the field sampling event is to collect data that are appropriate for developing and calibrating a QUAL2K model to simulate water quality constituents. The seven samples were collected along a 30,000 ft reach of the river. The sampling sites extend over the same spatial extent of the QUAL2K model.

One field study was performed for this project on August 31, 2021. Field work was performed in adherence to the Green Country Testing Quality Assurance Manual (QAM), see Attachment A. Water grab samples were collected (via boat) at the locations shown in Figure 1-2. Green Country Testing collected data regarding the pH, DO, temperature and specific conductivity of the water during the sampling collection. However, continuous in situ measurements were collected. Samples for all but Location #7 were collected in one day due to accessibility at the Lock and Dam #18. Location #7 was collected the following day (September 1, 2021). Due to the maintained and controlled nature of the Verdigris River, hydraulic measurements and flow data will be obtained from publicly available data sources (e.g., USACE flow data from Lock and Dam 18; USGS gage at Catoosa, Oklahoma). A second field study or model validation will not be performed.

2.1 Water Grab Samples

A total of 28 water grab samples were collected by boat over the seven locations, four samples at each location shown in Figure 1-2, for the constituents shown in Table 2-1. Composite samples were not collected. The grab sample collection standard operating procedure is included as Attachment B. Samples were collected as surface samples. A five-gallon bucket was placed in the Verdigris River, and water was allowed to flow into it to rinse the bucket back in the stream and letting water flow into it. The sample bottles were filled with the same mixture of water from the bucket at each location. The samples were put in an ice chest with ice before leaving the site and remained on ice until they are received by the laboratory. Green Country Testing provided clean sample bottles with labels and preservatives.

Parameter	Туре	No. of Samples	No. of Sampling Sites	Total No. of Samples
CBOD ₅ (Filtered)	Grab	4	7	28
CBOD ₅ (Unfiltered)	Grab	4	7	28
CBOD ₂₀ (Filtered)	Grab	4	7	28
CBOD ₂₀ (Unfiltered)	Grab	4	7	28
BOD ₂₀	Grab	4	7	28

Table 2-1: Field Samples

Parameter	Туре	No. of Samples	No. of Sampling Sites	Total No. of Samples
Total Kjeldahl Nitrogen – TKN	Grab	4	7	28
Ammonia Nitrogen – NH ₃	Grab	4	7	28
Nitrate – NO ₃	Grab	4	7	28
Nitrite – NO ₂	Grab	4	7	28
Total Phosphorus	Grab	4	7	28
Orthophosphorus	Grab	4	7	28
Enterococci	Grab	4	7	28
E. coli	Grab	4	7	28
Chlorophyll-a	Grab	4	7	28
Total Suspended solids	Grab	4	7	28
Volatile Suspended Solids	Grab	4	7	28
Total Organic Carbon	Grab	4	7	28
Dissolved Organic Carbon	Grab	4	7	28
Alkalinity	Grab	4	7	28

Prior to each field study, the buckets used for collecting stream samples were washed with detergent and rinsed with tap water and deionized water.

The samples analyzed for orthophosphorus were filtered within 15 minutes after the sample was collected. The materials and supplies used for filtering these samples in the field were membrane filters with 0.45 µm pore size, deionized water and a plastic syringe. The procedures used for filtering the orthophosphorus samples are included in Attachment B, and summarized below:

- 1. Pour approximately 5mL of deionized water into syringe and push it through to wash the filter.
- 2. Discard the filter wash water.
- 3. Pour sample water into syringe and push about 40-50 mL through the filter to fill the 40 mL vial.
- 4. Discard the used filter and any sample water not pushed through the filter. Rinse the syringe by pushing at least 50 mL of DI water through the syringe without any filter.
- 5. Repeat steps 1 through 4 to fill another 40 mL vial for each sample.

If the sample water contains so much suspended particles that the filter becomes clogged after pushing only 20-30 mL through it, the sample water can be pre-filtered using glass fiber filters. Pre-filtration

would be conducted using the same steps described above except using a glass fiber filter (more porous than a membrane filter). After prefiltering at least 125 mL, the pre-filtered water would then be filtered through a membrane filter.

2.2 Equipment Cleaning

As discussed previously, the only sampling equipment required beyond that provided by Green Country Testing was the five-gallon buckets. Prior to each field study, the buckets used for collecting stream samples or effluent samples were washed with phosphorus-free detergent and rinsed with tap water and deionized water. At each site, the buckets were rinsed once with ambient water before collecting a sample.

Prior to each field study, the equipment used for filtering the orthophosphorus samples were washed with detergent and rinsed with tap water and deionized water. At each sampling site, the syringe, and filter were washed by inserting the filter and pushing about 5 mL of deionized water through the syringe and filter. After completing filtration at a sampling site, the syringe was rinsed with deionized water.

No other field equipment was required to be cleaned or decontaminated during the field study.

2-3

3.0 QUALITY ASSURANCE & QUALITY CONTROL

The tasks and quality requirements for this project are designed to produce scientifically defensible recommendations for WLA limits of a point source discharge. The overall quality objective for achieving this scientifically defensible result is that the project design and the quality requirements for this project are consistent with EPA guidance and with previous WLAs and TMDLs that have been approved by EPA Region 6. If this quality objective is met, then the results of the project will be appropriate for their intended use as WLA limits. EPA's guidance for TMDLs is somewhat general due to the variability among TMDL projects; therefore, the EPA guidance does not address many details of a TMDL or WLA project. Those project details must be consistent with the overall objectives of the project.

Quality control (QC) activities in the field will include collecting field blank samples for the number of samples specified in Table 2-1. No other QC field samples (e.g., trip blanks, spiked samples) are necessary for this project.

The effectiveness of the control actions for sampling (replicates and blanks) will be evaluated after the analytical results are reported by the laboratory. If the holding time for an analysis has not expired, the project manager will instruct the laboratory to re-run the analysis if needed. Collecting a replacement sample from the field is usually not an option at this time. The effectiveness of these control actions will be reported to the client in the deliverable that documents the field data.

Quality control activities in the laboratories include analyzing laboratory duplicates, matrix spikes, and matrix spike duplicates following procedures and frequency of occurrence that are in accordance with each laboratory's standard QA/QC procedures. Procedures for quality control and quality assurance in the laboratory are discussed in Attachment A. The laboratory will follow this procedure to determine the exact types of QC samples that will be analyzed for this project and how many will be analyzed for this project. The project design and the quality requirements for this project are also consistent with, or more stringent than, other DO TMDLs and WLAs that have been approved by EPA Region 6. Below is more specific information of quality control as it relates to the work plan.

3.1 Field Data Collection

The performance criteria for field data are based on making sure that the field data can be used for calibrating and validating a water quality model that simulates DO and nutrients. The only purpose of the field data is to be used for the modeling. Those field data collected for this project are not intended for other uses such as assessing streams for 303(d) listing, evaluating long term water quality trends, comparing Verdigris River with other streams, or measuring storm loadings from different parts of the

watershed. The results of these field data will not change the project approach because these data will be used for the modeling (regardless of whether the data show high quality water or low quality water) and the modeling is a required part of the project approach.

These field data will be considered acceptable for calibrating and validating a water quality model if they meet the performance criteria listed below. These performance criteria address precision, bias, accuracy, representativeness, completeness, comparability, and sensitivity.

3.1.1 Precision

Precision for the laboratory analysis of the samples is discussed in Attachment A where the Green Country Testing QA/QC documentation is provided.

3.1.2 Accuracy

Accuracy for the laboratory analysis of the samples is discussed in Attachment A where the Green Country Testing QA/QC documentation is provided.

3.1.3 Bias

If the performance criteria for accuracy and precision are met, then any bias in these data will be considered acceptable. There are no procedures that can specifically evaluate bias of field procedures other than the ones already mentioned above for evaluating precision and accuracy.

3.1.4 Representativeness

There are no quantitative performance criteria for representativeness because it is a qualitative term. The three performance criteria for representativeness are as follows:

- 1. Data must be collected according to procedures outlined in this work plan for collecting samples and making various measurements
- 2. Data must be collected in locations that are not influenced by temporary or unrepresentative conditions such as construction at a bridge or trash dumped in the stream.
- 3. Each field study must be conducted when the conditions in the River are consistent with the following description. The first field survey should be collected near critical flow and temperature conditions. See Section **Error! Reference source not found.**

3.1.5 Completeness

Completeness of field measurements will likely be close to 100 percent. The performance criterion for completeness of each type of field measurements over the whole study area is 80 percent. If a sample is

not collected, that situation does not count towards incomplete data because valuable information is still being obtained (for water quality modeling, it is important to know if the stream is not flowing or is dry). If the completeness goal of 80 percent is met for each type of field data, there will be sufficient data for developing and calibrating the model.

3.1.6 Comparability

The performance criteria for comparability of field data collection areas follows:

- All field crews must collect data following the same procedures as outlined in this report or by standard procedures.
- All laboratory analyses for a given parameter must be conducted by the same laboratory using the same method.

Meeting these criteria will allow field data from different crews and different instruments to be combined into a uniform data set for this project.

3.1.7 Sensitivity

There is no field procedure for testing sensitivity. If all of the above objectives are achieved, the samples will be assumed to have acceptable sensitivity.

3.2 Laboratory Analysis and Sampling Handling/Custody

For the laboratory analyses QA/QC plan, the Green Country Testing QA/QC Manual will be utilized and is included in Attachment A. Other performance criteria for the laboratory analysis include following the correct chain of custody from the field to the lab. The chain of custody form will be kept in a large Ziploc bag inside the ice chest (on top of the ice and samples so that it can be retrieved easily). Each ice chest must have its own chain of custody form.

As mentioned previously, the laboratory will pre-label each sample bottle to identify the analyses that will be conducted with water from that bottle, and the field crew will label each sample with the site ID and the date and time the sample was collected. The site ID will be appended with "-B" for a field blank sample. When the lab receives the samples, they will log in each sample and assign it a control number or other unique identification.

At each sampling site, the field crew will put samples in an ice chest with ice before leaving the site and samples will remain on ice until the laboratory receives them. The field crew will replenish the ice in the ice chests to maintain ice prior to delivering samples to Green Country Testing. The temperature of the samples should be no more than 4°C when received by Green Country Testing.

analyses are shown in Table 3-1. This project does not have any action limits for the water quality data.

Revision 1

Parameter	Analytical Method	Preservation	Maximum Holding Time		
DO*]	ield Parameter - See SOP			
Temperature*	Field Parameter				
pH*	Field Parameter - See SOP				
Conductivity*]	Field Parameter - See SOP			
CBOD ₅ (filtered)	5210B, 2011	$Cool, \le 4^{\circ}C$	48 hours		
CBOD ₅ (unfiltered)	5210B, 2011	$Cool, \le 4^{\circ}C$	48 hours		
CBOD ₂₀ (filtered)	5210B, 2011	$Cool, \le 4^{\circ}C$	48 hours		
CBOD ₂₀ (unfiltered)	5210B, 2011	$Cool, \le 4^{\circ}C$	48 hours		
BOD ₂₀	5210B, 2011	$Cool, \le 4^{\circ}C$	48 hours		
Total Kjeldahl Nitrogen	4500 N _{org} B, 2011	Cool, $\leq 6^{\circ}$ C; H ₂ SO ₄ to pH <2	28 days		
Ammonia as N	4500 NH3-H, 2011	Cool, $\leq 6^{\circ}$ C; H ₂ SO ₄ to pH <2	28 days		
Nitrate as N	4500 NO3-F, 2011	$Cool, \le 6^{\circ}C$	48 hours		
Nitrite as N	4500 NO3-F, 2011	$Cool, \le 6^{\circ}C$	48 hours		
Total Phosphorus	4500 -P E, 2011	Cool, $\leq 6^{\circ}$ C; H ₂ SO ₄ to pH <2	28 days		
Orthophosphorus	4500 -P E, 2011	Filter immediately; Cool, $\leq 6^{\circ}$ C	48 hours		
Enterococci	Enterolert	Cool, $\leq 6^{\circ}$ C; 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours		
E. coli	9223B, 2004 or m- Coliblue-24	Cool, $\leq 6^{\circ}$ C; 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours		
Chlorophyll-a					
Total Suspended Solids	2540-D, 2011	$Cool, \le 6^{\circ}C$	7 days		
Volatile Suspended Solids	2540 B, 2011	$Cool, \le 6^{\circ}C$	7 days		
Total Organic Carbon	5310B, 2011	Cool, $\leq 6^{\circ}$ C; H ₂ SO ₄ or HCl or H ₃ PO ₄ to pH <2	28 days		
Dissolved Organic Carbon	5310B, 2011	$\begin{array}{c} \text{Cool}, \leq 6^{\circ}\text{C}; \text{H}_2\text{SO}_4 \text{ or HCl or} \\ \text{H}_3\text{PO}_4 \text{ to } \text{pH} < 2 \end{array}$	28 days		
Alkalinity	2320B, 2011	Cool, ≤ 6°C	14 days		

ATTACHMENT A- GREEN COUNTY TESTING QUALITY ASSURANCE MANUAL REVISION 29



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Quality Assurance Manual Page 1 of 36 Revision 029

Quality Assurance Manual

Green Country Testing, Inc.

Revision 029 Effective Date: 1/1/21 Supersedes Date: 2/10/20 Approved by:

Brian Duzan, Laboratory Director

Star Yuan, Quality Assurance Officer

Date

112512

REGISTRATION NUMBER: 651

REGISTERED TO: Laura Merriman

COMPANY: Burns and McDonnell OKC

DISPERSED: 1/25/21

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Quality Assurance Manual Page 1 of 36 Revision 029

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INTRODUCTION

The staff at Green Country Testing, Inc. (GCT) consists of highly qualified personnel having years of experience with the EPA methodologies. To service the needs of our customers better, our staff is actively pursuing the research and use of new methodologies and instrumentation. GCT provides full quality data packages on a standard turn time. Express service is also available without sacrificing data quality. In addition to our excellent turn time and extremely competitive fee schedules, a key to our success has been superior customer service. We at GCT understand the time pressures inherent to the environmental industry and have developed a management system to provide the highest quality results as rapidly as possible.

1.0 ORGANIZATION AND MANAGEMENT

1.1 Policy Statement

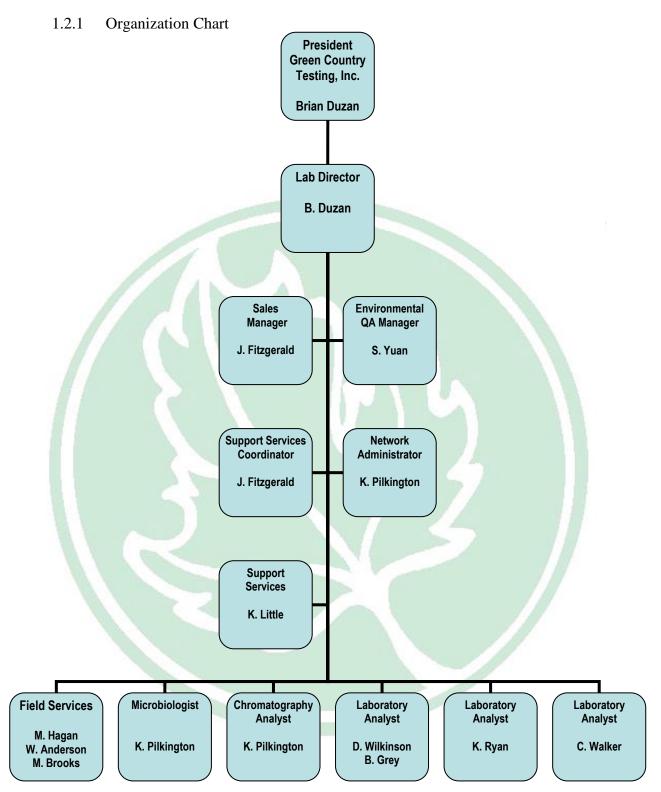
GCT has the responsibility and the commitment to establish procedures, which assure precise, accurate, complete, and representative data, is known and thoroughly documented. This Quality Assurance Manual contains the policies, procedures and requirements for the implementation and documentation of the quality assurance program used by GCT to ensure the production of scientifically sound, legally defensible data of proven quality. Holding times are only applicable to regulatory samples tested at GCT. All personnel concerned with environmental testing activities must familiarize themselves with the quality documentation and implement policies and procedures in their work. At GCT, Management has the highest commitment to compliance with The NELAC Institute (TNI), found in the 2009 TNI standard and ISO/IEC 17025:2005. The methodologies used by GCT's Environmental laboratory primarily include those published by the USEPA and other agencies in the texts listed in Appendix V of this document.

1.2 Organization

Green Country Testing, Inc. is a privately held corporation residing in Tulsa, OK and incorporated in the state of Oklahoma and is subject to all the laws of the state of Oklahoma and federal laws as required.

Executing an effective program demands the commitment and attention of both management and staff. The Laboratory Director manages the QA program. The implementation of the QA program is the responsibility of the QA Manager who reports directly to the President. The Group Leaders who reports to the Laboratory Director coordinates each laboratory department. In addition, all personnel within the organization play a vital role in assuring the quality of our work. We believe the success of Green Country Testing is dependent upon the continued commitment of all within the organization to a strong and viable QA program. For a complete list of laboratory personnel, See Appendix II.







1.2.2 Laboratories Capabilities

Gas Chromatography/Mass Spectroscopy (GC/MS) Section

GC/MS is an acronym for gas chromatography coupled mass spectrometry, the state-of-the-art instrumentation for the qualitative and quantitative assessment of low level organic pollutants. Analyses are performed on a wide variety of matrices including soils, waters, sludges, oils, and solvents. The environmental target analyses done by the GC/MS section include organic priority pollutants, EPA hazardous substance list (CLP list) and Skinner List (refinery residuals). The qualitative power of the GC/MS is routinely used to screen hazardous waste samples for disposal decision making.

Gas Chromatography (GC) Section

The chromatography section uses gas chromatographs (GC). By combining a variety of sample introduction techniques: on column injection, and purge and trap with selective and semi-selective detectors, sensitive and precise quantitation of many organic contaminants can be efficiently accomplished. The use of chromatographic separation techniques with selective detectors can achieve low parts per billion detection limits for many organic compounds of environmental concern. In addition to trace level environmental contaminants, the chromatography section performs the challenging task of characterizing waste streams for target organic constituents so that appropriate disposal procedures can be chosen.

Trace Metals Section

GCT's metals section has the capability to analyze samples for more than 70 parameters using both Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Atomic Absorption Spectroscopy (AA) technology. Samples that can be analyzed range from drinking waters to soil and hazardous wastes.

General Chemistry Section

The general chemistry section of the laboratory has capabilities for over forty different analyses making GCT truly a full service laboratory. A variety of methodologies are employed to test water, wastewater, soil and hazardous waste. The general chemistry section is responsible for classical, physical, and colorimetric analysis of environmental samples as well as more sophisticated analysis using TOC. The analytical information developed in this and other sections is necessary for appropriate waste disposal procedures to be followed.

Microbiology Section

The microbiology section of the laboratory has capabilities for various microorganisms including Total Coliform, Fecal Coliform, E. Coli, and Enterococcus. The analytical information developed in this and other sections is necessary for appropriate compliance.



Data Management Section

The data management section and the support services group are responsible for sample custody in the laboratory, sample log in, sample tracking, and accounting and report generation. Data management maintains and operates the laboratory's custom developed computerized Laboratory Information Management System. Data management is the heart of analytical flow and sample management. For maintenance and operational procedures, refer to GCT LIMS Training Manual.

Field Services Section

The field services section within the support services group provides pick-up services in the surrounding area. This section also performs manhole, tank, drum, soil, and NPDES sampling.

Network Administration Section

The IT section is responsible for the integrity of the GCT website, the server and the network of computers related to all above stated sections. The IT section is also responsible for keeping the LIMS system in pristine working condition. This LIMS system tracks all aspects of samples and their complete process through the lab including reagents and chemicals related to their analysis. In addition, the IT section is accountable for data archival and its storage.

1.2.3 Laboratory Personnel, Authority and Responsibilities

Laboratory Director

Minimum Requirements

- Bachelor's Degree in Science or 4 years experience in a related field
- 24 Hours of College Chemisry
- 5 years experience in the area of environmental analysis and field of accreditation
- The Laboratory Director is also the Technical Director Responsibilities
- In charge of day to day operations
- Monitoring the QA Plan in coordination with the QA Manager to ensure all data generated is scientifically sound, legally defensible, and of known precision and accuracy
- Commitment to the development and implementation of the QA system
- Ensuring compliance with various state and federal agencies.
- Ensuring compliance with 2009 TNI Standard and the International Standard ISO/IEC 17025:2005.
- Prescribing and monitoring corrective actions
- Coordinating certification programs within the laboratory
- Scheduling and conducting annual management audits
- Ensuring appropriate communication between all parties within the laboratory

Authority

The Director is the final authority on all issues dealing with data quality and has the authority to require that procedures be amended or



discontinued, or analyses suspended or repeated. He also has the authority to suspend or terminate employees on the grounds of dishonesty, incompetence, or repeated non-compliance with QA procedures.

Deputization

In the absence of the Director or QA Manager, QAO, Star Yuan shall hold responsibility for all technical decisions and interpretations involving testing procedures, methods, and reports. In the event that the Technical Director has a temporary absence of 15 consecutive days, Star Yuan shall be the designated, qualified staff member taking this role. The accrediting body shall be notified in writing in the event that the Technical Director is absent for 65 consecutive calendar days. Keli Pilkington has been designated as the Head over the Microbiology Department at Green Country Testing, Inc. The laboratories approved signatories shall be Brian Duzan and Star Yuan. In the event that an approved signatory signs a report, it shall be indicated who the report is signed for. This applies to all reports in the old LIMS system. The new system reports are all sent electronically via email.

Quality Assurance Manager

Minimum Requirements

- Bachelor of science degree or 4 years experience in a related field
- Two years experience in environmental testing
- Knowledge and understanding of all methods being performed in the laboratory
- Knowledge and understanding the NELAC Quality System. Responsibilities
- Developing and implementing QA procedures within Green Country Testing to improve and maintain consistent data quality
- Ensuring compliance with 2009 TNI Standard and the International Standard ISO/IEC 17025:2005.
- Maintaining a current version of the QA plan and annually reviewing the QA plan to assess and to ensure the effectiveness of the QA program
- Maintaining other laboratory procedures.
- Maintaining records and archives of all QA/QC data, PT results, audit comments, and customer inquiries concerning data quality.
- Performing statistical analyses of QC data and establishing data bases which accurately reflect the performance of the laboratory
- Distributing performance evaluation (PT) samples on a routine basis
- Evaluating the results of PT samples, and applying corrective actions as needed to ensure each department is able to generate data which meets the data quality objectives defined in this QA plan
- Reporting the status of the laboratory QA program to the Director with formal and informal communications

<u>Authority</u>

The Quality Assurance Manager of the laboratory will be independent from laboratory operations. The Quality Assurance Manager of the



laboratory has the authority to stop work for reasons of doubtful quality and, in conjunction with the Director, can accept data that fall outside normal QC limits if, in their judgement, there are technical reasons that warrant the acceptance of the data, but the data will still be flagged accordingly. The QA Officer will have access to the highest level of management for any decisions involving the quality of data or the quality program.

Deputization

In the absence of the QA Manager, the Group Leader of each test area shall hold responsibility in their respective testing divisions to accept or reject data based on well-defined QC criteria.

Chromatography Analyst

Minimum Requirements

- Bachelor degree and/or
- Two years experience in environmental testing

Chemists

Minimum Requirements

- Bachelor of science degree
- One year experience in environmental testing

Technicians (Analysts)

Minimum Requirements

- High school diploma or equivalent
- Six months experience in environmental testing <u>Responsibilities</u>
- Having a working knowledge of the Green Country Testing's QA plan
- Ensuring all work is generated in compliance with the Green Country Testing's QA plan
- Performing all work according to defined methods
- Ensuring all documentation related to their work is complete and accurate

• Providing management with immediate notification of quality problems <u>Authority</u>

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC acceptance criteria. Laboratory management must approve the acceptance of data that falls outside QC criteria.

Network Administrator

Minimum Requirements

- High school diploma or equivalent
- Knowledge in Server 2003, MS Access and Office suite, TCP/IP, HTML Responsibilities
- Imaging and restoration of workstations
- Archiving and backup of data
- Updating of the GCT website



• Maintenance of all equipment in the GCT.intra network and LIMS system

Support Services

Minimum Requirements

- High school diploma or equivalent
- Working knowledge of DOS and Windows

<u>Responsibilities</u>

- Sample receipt and login
- Checking for proper sample containers, collection and preservatives
- Distributing samples throughout the laboratory for analysis
- Client services

Support Services Coordinator

Minimum Requirements

- High school diploma or equivalent
- Working knowledge of DOS and Windows
- **Responsibilities**
- Client services
- Printing of draft and final reports
- Maintaining the LIMS system
- Scheduling of sampling events

Field Services

Minimum Requirements

- High school diploma or equivalent
- **Responsibilities**
- Collecting and pickup of samples
- Preparing sampling containers and equipment
- Field testing of parameters with short holding times such as pH and Total Residual Chlorine.

Sales Manager

The Sales Manager position is a non-technical position. The requirements for this position will be evaluated on a case by case basis.

1.2.4 Client Confidentiality and Conflict of Interest

Green Country Testing's policy regarding client confidentiality is very rigid. Only authorized personnel may give verbal results. GCT personnel may only give results to other individuals if GCT has received prior consent from the client. Test data shall only be supplied to the client submitting the sample. Only at the request of the client shall information be released to a third party. This request shall be submitted in writing, where applicable. Only authorized employees are allowed access to a customer's file.



All GCT employees are obligated to maintain all company trade secrets, proprietary information, and confidential information. Confidential information includes, without limitation, manufacturing processes, computer programs, financial data, and marketing plans. Management and personnel are to be free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work.

All proprietary information received by a customer shall be held in strict confidence and will not be used for any purposes other than provision of the services for Green Country Testing. Employees shall not copy, transmit, publish, summarize, quote, or make any commercial or other uses whatsoever of Green Country Testing or customer confidential information.

All Green Country Testings employees are obligated to maintain objectivity and be free of conflicts of interest in discharging their professional responsibilities. Conflicts of interest include, but are not limited to, internal pressures, acquisition or commitment to acquire any direct or material indirect financial interest in a customer and/or participation as a promoter, voting trustee, director, or Manager of a client. Violation of this trust will result in disciplinary action and any other remedies provided by applicable law.

1.2.5 Laboratory Feedback

Green Country Testing, Inc. actively engages in feedback from the customers. This could be informal conversations or detailed question and answers. The general information will be recorded in the Client Conversation Log in Omega. If a complaint comes up, it will be recorded in the Customer Complaint Log. If corrective action or other action is to be taken resulting in the conversation, the appropriate GCT personnel will be informed and the appropriate steps will be taken.

2.0 AUDITS, ESSENTIAL QUALITY CONTROLS AND DATA VERIFICATION

2.1 Internal Audits

It is the responsibility of the QA Manager to plan and organize internal audits. All areas of the laboratory will be audited annually by qualified personnel who are, wherever resources permit, independent of the activity to be audited. Internal audits identify sources of potential nonconformances and are proactive measures to improve the QA program. Areas will be audited at a rate of one per month. Areas will be audited in a manner such that all analysts will be audited annually. If a client's work is affected by an Internal Audit, they will be notified in writing within one month. When an internal or third party audit takes place and non-conformances arise, corrective action will be instituted.

The following 12 areas shall be scheduled for internal audit during each year. A review shall be scheduled when there is evidence supporting data integrity issues. Client notification of the corrective action is required when these issues arise. The Quality



Assurance Officer will conduct an Internal Audit on all employees annually or sooner if there is any evidence of inappropriate actions or vulnerabilities related to data integrity.

The areas are Login, Subcontracting, Oil and Grease, Trace Metals, Solids, BOD, Wet Chemistry, Soils, GC, GCMS, Field, Microbiology. Archived records, customer service, and the IT department are also reviewed in addition to these topics. Quality Control and Misc. items are also included.

- 2.1.1 As part of the internal audit schedule an audit trail review in Omega will be conducted every six months. In March and September the Quality Officer will review and document the audit trail data. Every six months when the audit trail is reviewed, the previous six months will be looked at.
- 2.1.2 As part of the internal audit schedule, a semi-annual review of holding time reports will be conducted. In March and September the Quality Officer will review and document the holding time report data for various tests in the laboratory.
- 2.1.3 Additional audits may be required from time to time. Additional audits may result from an outside audit, corrective action, anonymous tip, or other action. The QA Officer may use whatever form is adequate to detail the information.

2.2 Management Review

Management review is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. The QA Manager will submit reports to management summarizing the information gathered throughout the laboratory. The review will take into account the following:

- 1. Report and discussion of matters arising from the last meeting.
- 2. Report and discussion of any correspondence from accrediting agency.
- 3. Discussion of client audit reports.
- 4. Results of internal audits with appropriate corrective action.
- 5. Results from participation in proficiency testing.
- 6. In house quality checks.
- 7. Client complaints.
- 8. Personnel including issues and training for new and existing staff.
- 9. Adequacy of staff, equipment, facilities.
- 10. Future plans and estimates for new work, staff, equipment, etc.
- 11. Suggestions for improvement of quality system.
- 12. Preventive Actions.
- 13. Corrective Actions.
- 14. The Suitability of policies and procedures
- 15. Other concerns impacting the quality system.
- 16. Actions to be taken as a result of the Annual Management Review.

The Annual Management Review will be assessed on a pre-determined schedule. This review shall be done within the first 30 calendar days of the year. During the annual management review, data integrity procedures shall be reviewed and approved by management.



2.3 Audit Review

All audit and review findings and any corrective actions that arise from them will be documented and those documents maintained in the QA files.

2.4 Proficiency Testing (PT) Program

Green Country Testing operates its PT program under the "Run one, Report one" philosophy. All PT samples are handled exactly like client samples. The laboratory participates in two PT studies per year per matrix, where applicable. Requirements are the satisfactory completion of two PT studies out of the 3 most recent PT studies attempted at a frequency of two PT studies per year, approximately six months apart. Results are considered satisfactory when they are within the acceptance limits established by the vendor. These results will be shared with certifying agencies along with the appropriate corrective action including the results of a Corrective Action PT sample. Please refer to the SOP on PT Studies for more detailed information.

2.5 Preventive Actions

Preventive action is a pro-active process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints. The laboratory shall identify needed improvements and potential sources of nonconformances, either technical or concerning the quality system. If preventive action is required, action plans shall be developed, implemented and monitored to reduce the likelihood of the occurrence of such nonconformances and to take advantage of the opportunities for improvement. The following are routine functions performed as preventive actions to identify improvements in the quality system:

- 1.) Internal Audits-Every month the QA/QC Officer will audit an area of the laboratory to identify sources of non-conformances and other issues leading to improvements in the quality system. For more information on internal audits refer to section 2.1 of the QAM.
- 2.) Control Charts-Twice a year the QA/QC Officer updates control charts in an effort to identify potential problems or non-conformances, and to track historical data generated by the laboratory. For specific information on control charting refer to the SOP on control charts.
- 3.) PT Studies-Twice a year the laboratory analyzes blind PT's to identify any analytical problems in the quality system. For more information on proficiency testing refer to section 2.4 of the QAM.
- 2.6 Nonconforming Work

When any aspect of the testing and/or calibration and/or results do not conform to the laboratories procedures (QAM and SOP's) or the agreed requirements with the customer, this shall be determined to be non-conforming work.

2.6.1 Arriving Samples



When samples arrive at the laboratory and the requested analysis can't be performed (i.e. hold time already expired, improperly preserved, not enough sample), the client shall be notified. The client may request the samples to be analyzed anyway, but the data will be qualified.

2.6.2 During Analysis or Already Analyzed Data

The Laboratory Director or Quality Assurance Officer will be notified immediately to evaluate the significance of the nonconforming work. Once evaluated, action will be taken immediately including the possibility of stopping work, withholding of test reports, notifying the customer, recalling reports, or amending reports. Nonconforming work may occur at various places within the management system including customer complaints, quality control, instrument calibration, consumables, staff observations, management reviews, internal audits, or external audits. Resumption of work will not begin until the issue has been resolved and the effected test is in conformance to the laboratories written procedures.

When the evaluation indicates that the nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, corrective action will be initiated.

2.7 Corrective Actions

To the extent possible, samples will be reported only if all quality control samples within the batch are acceptable. If a quality control measure is found to be out of control and sample reanalysis cannot be performed due to insufficient sample, the data will be reported and all samples associated with the failed quality control sample(s) will be flagged. Notations of failed quality control samples will be made to the Case Narrative. Green Country Testing, Inc. shall notify the primary Accrediting Bodies of any corrective actions taken for failed PT samples within 30 calendar days after the receipt of the final report from the PT provider. For any 2 out of 3 failed PT samples a Quik Response shall be obtained. The responsibility for authorizing the resumption of work following the finding of a nonconformance is the decision of management at GCT.

For more detailed information, refer to the Corrective Action SOP.

2.8 Essential Quality Control

2.8.1 Precision:

The degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of reference materials, environmental samples, or laboratory control samples. Green Country Testing routinely monitors precision by comparing the %RSD (relative standard deviation) of RPD (relative percent difference) between check sample measurements with control limits established at plus and minus three standard deviations from the mean RPD of historical check sample data.



2.8.2 Accuracy:

A determination of how close the measurement is to the true value. Accuracy can be assessed using standard reference materials, check samples, or spiked environmental samples. Unless specified otherwise in special contracts, Green Country Testing monitors accuracy by comparing check sample results with the control limits established at plus/minus three standard deviation units from the mean of percent recovery of historical check sample results. The determination of the accuracy of a measurement requires knowledge of the true or accepted value for the analyte signal being measured.

2.8.3 Completeness

A measure of the amount of valid data obtained from a measurement system compared with the amount that is expected to be obtained under correct normal conditions. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the database is sufficient.

2.8.4 Detection Limits

Method Detection Limit (MDL)

An MDL shall be initially determined for the compounds of interest in each test method in a quality system matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the MDL must be determined in the quality system matrix of interest.

An MDL is the minimum signal level required to qualitatively identify a specific analyte by a specific procedure at 99% confidence level. An MDL is measured by analyzing a minimum of seven (7) replicates spiked at one (1) to five (5) times the expected method detection limit. It is calculated by the student t-value (99%) with n-1 degree of freedom times the standard deviation (MDL = t (n-1, 1a=0.99)(s), where t (n-1, 1-a=0.99) is the t-statistic appropriate for the n number of samples used to determine s, at the 99% level. The LOD may also be determined by using blank data, but this process is not approved by the Oklahoma Department of Environmental Quality for MDL studies. Green Country Testing chooses to use the above procedure to determine MDL's as documented in 40 CFR Part 136 Appendix B. If a method has a specific process for conducting a detection limit study, that process will be followed for that method and documented accordingly. An MDL study is not required for any component for which spiking solutions or quality control samples are not available. The MDL must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

Limit of Detection (LOD)

If the laboratory reports lower than the PQL or as method specified, an LOD must be determined. If the laboratory does not report below the PQL or as specified in the method, an LOD does not need to be determined. The validity of the LOD shall be confirmed by qualitative identification of the analyte(s) in a QC



sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests and 1-4X the LOD for multiple analyte tests. This verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data. The LOD shall be verified annually for each quality system matrix, method, and analyte.

Limit Of Quantitation (LOQ) - Practical Quantitation Limit (PQL).

The <u>LOQ / PQL</u> shall be at level greater than the MDL. The LOQ/PQL study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate. The validity of the LOQ/PQL shall be confirmed annually by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2X the claimed LOQ.

2.8.5 Estimation of Uncertainty of Measurement

In most cases the nature of the test method will preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. As outlined in A2LA's "Guide for Estimation of Measurement Uncertainty in Testing", the best over all process is to use control charting. By control charting a known or accepted value, we can set the action limits for an estimate of measurement uncertainty at one standard deviation. The control charts can be generated upon client's request.

For more detailed information, refer to the various analytical SOPs and the SOP on Control Charts.

2.8.6 Method Blanks

Reagent or analytical blanks are analyzed to assess the level of contamination that exists in the analytical system and might lead to the reporting of elevated concentration levels or false positive data. An analytical blank is analyzed with every batch of samples processed. The analytical blank consists of reagents specific to the method carried through every aspect of the procedure, including preparation, clean up, and analysis. The Method Blank will be matrix matched to the samples. If water samples are analyzed, a water MB will be analyzed. If a soil sample is analyzed, glass beads, Ottawa sand, or other solid matrix will be used. Ideally, the concentration of an analyte in the blank is below the reporting limit for that analyte. However, some common laboratory solvents and metals are difficult to eliminate to the parts-per-billion level commonly reported in environmental analyses. If the MB is found to be contaminated, the batch will be reprocessed or the resulting data flagged accordingly. Documentation shall be provided by the analyst in the appropriate logbook each time a method blank is contaminated. Reprocessing or data qualification may be necessary. Upon data review by the QA/QC officer corrective action shall be necessary to minimize or eliminate the problem of contaminated blanks. Method Blanks are not required for those tests where it is not applicable (pH, temperature, etc...).



2.8.7 Laboratory Control Samples

For those analysis that do not require a preparation step, the ICV and the LCS can be considered the same thing and will be prepared from a separate NIST traceable source than the standards. For those analysis that require a preparation step, the LCS can be from the same source as the standards. The LCS will be matrix matched to the samples with a similar volume as the samples. If water samples are analyzed, a water LCS will be analyzed. If a soil sample is analyzed, glass beads, Ottawa sand, or other solid matrix will be used. LCSs are used to monitor performance of routine analytical methods on a day-to-day basis. They are analyzed with each batch of samples and are supplemented with surrogates (where methods require) to ensure operational QC is available with each batch of samples processed. For those methods that allow it, a matrix spike may be used in place of an LCS. For those methods where spiking solutions are not available, LCS's may not be required (pH, temperature, dissolved oxygen etc...). For those tests that have multiple analytes, the laboratory shall spike all target compounds at least every two years. It is our general policy to spike for every compound with every batch to fully evaluate the quality system.

- 2.8.7.1 If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside the control limits. This may not indicate the system is out of control, therefore corrective action may not be necessary. Upper and lower marginal exceedence (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.
- 2.8.7.2 The number of analytes in the LCS is the basis for the number of allowable marginal exceedances. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. For methods with long lists of analytes, the marginal exceedance approach is relevant. It will not apply to target analytes lists with fewer than 11 analytes.

The number of allowable marginal exceedances is as follows:

- >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit;
- 2) 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit;
- 3) 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit;
- 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit;
- 5) 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit;
- 6) <11 analytes in LCS, no analytes allowed in ME of the LCS control limit;



Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. Corrective action must be taken after the source of the error is located

2.8.8 Duplicates

A duplicate is a sample that is divided into two separate aliquots. The aliquots are processed separately and the results (expressed as relative percent difference [RPD]) are compared to determine the effects of the matrix on the precision of the analysis.

2.8.9 Matrix Spike and Matrix Spike Duplicates

A matrix spike (MS) is a routine sample to which known concentrations of analytes have been added. The frequency of analysis of MS and MSD's is specified in the SOP's unless client specifies additional use of spikes. The MS is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis. A matrix spike duplicate (MSD) is a sample divided into two separate aliquots, each of which is spiked with known concentrations of analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery. For those tests that have multiple analytes, the laboratory shall spike all target compounds at least every two years. It is our general policy to spike for every compound with every batch to fully evaluate the quality system.

- NOTE: If the spike concentration is not specified in the SOP or if the sample exhibits unusual characteristics, the analyst may use discretion as to what level is necessary during analysis.
- 2.8.10 Surrogate Recoveries

Green Country Testing routinely adds surrogates to samples requiring GC analysis. These surrogates are organic compounds that are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. Surrogates will be added to all samples, standards, and QC samples if appropriate. The surrogate recoveries are used by the lab to assess both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix. Surrogate recovery information is routinely reported to clients with all data packages.

2.8.11 Initial Calibration Verification

Second source certified and/or NIST traceable standards are used with each routine analytical method. For those analysis that do not require a preparation step, the ICV and the LCS can be considered the same thing. For those analysis that require a preparation step, the ICV is prepared as the standards are but from a different source.



2.8.12 Demonstration of Capabilities

Initial Demonstration of Capabilities

Prior to using a method for the first time or if there has been changes in instruments, personnel or methods or if the method has not been performed by the laboratory or analyst in twelve months, or adding additional analytes to a previously accepted method, an Initial Demonstration of Capabilities will be performed and passed. For those testes where a method has been use for more than a year prior to seeking accreditation, and there has been no significant changes to instrument or personnel, the on-going demonstration of capabilities shall be satisfactory, and the laboratory shall retain all pertinent data. All DOC's shall be fully documented including analyst, matrix, method, analytes, SOP or method, prevision number, summary of data, calculations of data, acceptance criteria, any other information that is important.

If the method contains a specific procedure for the IDOC's, it will be followed. If the method does not contain a specific procedure, the following procedure will be used. The analytes shall be diluted in a clean appropriate matrix free of interferences at the appropriate level. If no level is specified, one to four times the LOO / POL may be used as a guideline. At least four aliquots shall be prepared and analyzed according to the method either concurrently or over a period of days. All preparation and analytical steps must be completed for the DOC. Using the data from all of the results, calculate the average and standard For those tests where it is not possible to conduct statistical deviations. evaluation of the data, the laboratory shall assess performance against established or documented criteria. Compare the results to the acceptance criteria for precision and accuracy in the method if available. If the method does not have acceptance criteria, laboratory generated criteria will be used. If all parameters pass, analysis may begin. If any parameter fails, the procedure must be repeated for that parameter until it passes. Repeated failure confirms a general problem in the measurement system, and it must be located and corrected before moving forward.

On-Going Demonstration of Capabilities

Annually, an on-going Demonstration of Capabilities shall be performed. It may be documented by the use of an acceptable blind performance study (PT), four LCS's that meet the acceptance criteria, or another IDOC. If the method states specific processes or acceptance criteria, the method information shall be used. All DOC's shall be fully documented including analyst, matrix, method, analytes, SOP, SOP or method revision number, summary of data, calculations of data, acceptance criteria, any other information that is important.

3.0 PERSONNEL

3.1 Laboratory Management Responsibilities

The quality of all tests performed by Green Country Testing is dependent to a great extent on the skill levels and manpower distribution of all operating personnel. The QA



department shall review each new program for special requirements, and periodically thereafter review the test capabilities of the equipment and the personnel.

Minimum Training Requirements include the following:

Instrument Operators1 year experience + trainingAnalyst - Chemistry Procedures6 months experience + trainingAnalyst - Microbiological Procedures6 months experience + trainingSample Preparation3 months experience + trainingSample Collection and Field Testing6 months experience + training

Technicians in training must demonstrate the ability to reproduce reliable results through in-house QC samples and proficiency testing. They must work directly under the supervision of the Director, QA Manager, Technical Manager or Group Leader.

3.2 Records

The QA Manager maintains training files on every analyst. This file includes the analyst's education and experience credentials, training certificates, records of training courses, safety courses in compliance with OSHA guidelines, and analyst proficiency testing results. Training is provided for all personnel on an on-going basis.

4.0 FACILITIES – ACCOMODATION AND ENVIRONMENT

4.1 Environment and Work Areas

All work will be conducted at the laboratories permanent location at 6825 E. 38th Street, Tulsa, OK 74145 except those tests that can be conducted in the field (pH, Temperature, Total Residual Chlorine, Dissolved Oxygen, etc...). The laboratory is considered a controlled area. Routine visitors (compressed gas deliveries, DI water maintenance, etc...) do not require escort. Any non-routine visitors (salesmen, clients, etc...) shall be escorted by GCT personnel. Test equipment and standards shall be calibrated and utilized in an environment controlled to the extent that biological sterility, dust, electromagnetic disturbances, radiation, humidity, electrical supply, temperature, sound, vibration, and corrosion or other factors do not interfere with precision measurements. If an environmental problem arises that affects the tests or calibrations, the test shall be stopped and the situation evaluated before continuing. The conductivity of the Type II water used by the laboratory is checked weekly and recorded.

The laboratory enforces a physical separation of preparation or analysis when such activities would adversely affect the quality of the testing or measurement including cross-contamination. Special procedures may be necessary to ensure quality testing or calibration. When required for equipment accuracy, certain testing areas shall be monitored for temperature and humidity control. Good housekeeping efforts will be employed at all times.



4.2 Evaluation of New Work

Upon the review of requests, tenders, and contracts at GCT, the laboratory shall ensure that the policies and procedures for these reviews leading to a contract for testing shall be defined, documented and understood. All new work shall be initially reviewed by customer service personnel. This review is more geared to gathering information for managements review. The customer service personnel will gather from the client there basic information (e.g., address, telephone), the nature of the project (e.g., city permit, monitor wells), tests and methods, programs that they have to follow (e.g., NPDES, RCRA), how they want the data reported to them and to whom the data is to be sent. The Laboratory Director will then take the collected information and use it to determine if the laboratory has the appropriate facilities, resources, and certifications available before accepting samples. The Laboratory Director will maintain a logbook documenting the review of requests, tenders, and contracts. The Laboratory Director will record the Client, if it is a new client, if it is a contract, if it is for compliance, if it has any unusual requirements, the analysis requested, and if our certification covers the requested analysis.

In the event that work has commenced and a new contract needs to be amended, the amended contract will be reviewed to ensure it meets all the necessary requirements. The information from the amendment will be relayed to all affected personnel. Records of reviews, including any significant changes, shall be maintained. Records shall also be 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. These records which relate to pertinent, quality objectives shall be maintained in various client conversation logbooks throughout the laboratory or in the LIMS system in the client conversation phone log.

A contract may be any written or oral agreement to provide a client with environmental testing services. Any differences between the request or tender and the contract shall be resolved before any work commences. Each contract shall be acceptable by both the laboratory and the client.

In the event that the laboratory suffers a suspension or loss of accreditation, clients will be notified in writing via our customer service department within 7 working days.

5.0 EQUIPMENT AND REFERENCE MATERIALS

Any instrument which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by verification or otherwise to be defective, shall be taken out of service, clearly identified and wherever possible stored at a specified place until it has been repaired and shown by calibration, verification or check standard to perform satisfactorily. If the equipment is sent off-site for repair or outside direct control of the laboratory, it shall be checked by the laboratory prior to being put back in service. The laboratory shall examine the effect of this defect on previous tests, and where necessary reanalyze questionable samples.

Each major instrument shall be labeled, marked or otherwise identified to indicate its calibration status. Records are maintained on each major piece of equipment and are



located in the Equipment Manual and QA files. Manufacturer's manuals are kept in various locations throughout the laboratory to enable convenient access. Maintenance logbooks are utilized in the LIMS or in paper form to document the details of maintenance carried out to date and planned for the future, as well as, history of any damage, malfunction, modification or repair.

5.1 New Equipment

The Director, Technical Manager or QA Manager is responsible for establishing and maintaining a system for the qualification and calibration of all new test and measuring equipment used for testing. All test equipment shall be given a unique GCT Asset Tag Number. This unique identification shall be used in logbooks and in the LIMS to track equipment usage. All new equipment shall be installed, qualified, and calibrated with the use of a check standard, or by certifiable standards prior to use. All test and measuring equipment shall be recalibrated at regular intervals or whenever subjected to an environment that may affect its accuracy.

5.2 Equipment Traceability

All equipment will be given a unique GCT Asset Tag Number. A master list will be maintained by the Laboratory Director that also gives the manufacturers serial number, if available. All equipment and operating conditions will be able to be traced.

5.2.1 Operating Conditions

All operating conditions are detailed in the appropriate analytical SOP. The analyst is not allowed to deviate from the prescribed operating conditions without approval from the laboratory director. Deviations must be documented and data qualified accordingly.

5.2.2 Traceability

All equipment and instruments used during the analytical process including pH meters, pH probes, rotators, thermometers, GC's, balances, or any other piece of equipment shall be traceable using either the analytical logbook or the LIMS. Some tests list all the equipment within the analytical logbook by the GCT Asset Tag Number or manufacturer's serial number. Some tests utilize the LIMS. All the equipment and instruments are listed in the LIMS. Equipment Sets are generated for a test and include all pertinent information. The Equipment Set is automatically linked to each run log in the LIMS for any given test. This allows for very detailed information to be documented easily.

5.3 Reference Materials

Records of reference materials significant to the tests performed are maintained in the form of certificates of analysis. Material will be traceable to NIST or other acceptable level and will be traceable to SI units. If any internal reference materials are utilized, they will be checked for technical and economical practicability. All of these records



are stored in the QA files. Please refer to the various analytical SOP's on standard preparation, safe handling, transport and storage.

6.0 MEASUREMENT TRACEABILITY AND CALIBRATION

6.1 General Requirements

The laboratory shall have available all the necessary equipment, reference material, and methodologies to perform all analyses in accordance with US EPA protocols. All measuring operations and testing equipment having an effect on the accuracy or validity of tests shall be calibrated and/or verified before being put into service and on a continuing basis and checked by authorized personnel.

All test equipment shall be operated in accordance with written test procedures. In the event the laboratory must use equipment outside its permanent control, it shall ensure that the relevant requirements are met. All equipment shall be properly maintained. Maintenance procedures are documented in this manual and in the various equipment SOPs.

6.2 Traceability of Calibration

All calibration standards are traceable to national standards of measurement. Calibration records include the lot number of the appropriate standard(s). It is the QA Manager's responsibility to maintain records of all certificates of analysis for each lot of standard or reagent purchased by the lab. Records are maintained for at least 10 years or the life of the material, whichever is longer.

6.3 Reference Standards

Reference standards of measurement such as Class 1 weights or NIST traceable thermometers are used for calibration or verification ONLY and no other purpose. This equipment is calibrated annually by certified calibrating bodies or in-house by Green Country Testing personnel. Calibration certificates are kept in the LIMS. The laboratory will conform to all manufacturing guidelines and procedures for the safe handling, transport, storage and use of reference standards in order to prevent contamination and protect their integrity.

6.4 Support Equipment

Support equipment is the necessary equipment used to support laboratory operations. These include but are not limited to: environmental chambers, balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. Ovens, water baths, incubators, refrigerators, and similar equipment are usually set to a specific temperature based on method requirements. The staff shall not change the conditions without



approval of the QA Officer or Laboratory Director to verify that the change does not affect method or quality criteria.

All support equipment shall be maintained to ensure that the equipment is capable of being properly calibrated at least annually to NIST or other traceable references, bracketing the range of use where applicable. See **various SOP's** for calibration acceptance criteria. If correction factors are to be used, the records will be maintained by the QA officer.

When support equipment can't be properly calibrated, the QA Officer or Laboratory Director shall be notified immediately so that the equipment can be removed from service. Service reports are kept in the LIMS or within the lab containing the equipment. Maintenance logs are kept in the LIMS and will detail maintenance activities including service calls. For field equipment, a logbook page will be inserted in the back of the analytical logbook so that maintenance can be recorded in the field. After return to the laboratory, the information shall be scanned into the LIMS. If the support equipment falls outside of the acceptable allowances and compliance samples were involved, (i.e. an incubator with samples in it over the week-end exceeds the minimum or maximum allowance), a corrective action will be initiated to determine not only the cause of the failure but to also qualify any data, if required.

Equipment will be calibrated or verified in their range of use and the raw data retained on the following schedule:

Autoclaves	daily or before each use by analyst using fixed
	thermometer + indicator
Balances	daily or before each use by analyst + annual service by
	certified vendor
Conductivity meters	daily or before each use by analyst
Freezers	daily $\leq 0^{\circ}$ C by analyst
Fume Hoods	quarterly by QA Manager or designee
NIST traceable thermometers	monthly by QA Manager or designee
Ovens, Incubators, and Baths	daily or before each use by analyst
Total Coliform & Fecal Coliform In	cubators twice daily at least 4 hours apart
pH meter	daily or before each use by analyst
Pipettors (Eppendorf or Repipets)	quarterly by QA Manager or designee
Refrigerators	daily (0-6°C)(may vary if method specific) by analyst
Working thermometers	annually by QA Manager or designee
Re-pipettors	quarterly by QA Manager or designee
Non-"Class A" glassware	once prior to use and documented accordingly
Bar Code Readers	once prior to use and documented accordingly

6.5 Instrument Calibration



The details of initial instrument calibration are included in the method Standard Operating Procedure (SOP). Sufficient raw data records are retained to permit reconstruction of the initial instrument calibration including calibration date, test method, instrument, analysis date, analyte name(s), analyst, concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration.

Initial calibrations are verified with a second source standard (ICV or LCS) or a lot other than that of the calibration standard and meet the appropriate acceptance criteria. In the case that testing includes sample preparation, the analyst shall analyze the second source standard without preparation. All calibration standards must be traceable to national standards, where available. Certificates of analysis for calibration standards are kept in the QA files.

Method specified acceptance criteria will be followed. If the method does not specify acceptance criteria, the calibration curves will require a minimum of 3 standards. Acceptable calibration curves require a 0.995 or better correlation coefficient. If this criteria is not met, the problem must be identified and resolved before the calibration is attempted again. If the calibration is automated and samples are analyzed with it and the acceptance criteria is not met, corrective actions must be performed, documented, and the calibration and all samples reanalyzed. For those tests where only a zero point and a single point calibration is employed, a standard at or below the LOQ /PQL shall be analyzed every day the instrument is being used, and it shall pass the appropriate acceptance criteria.

Sample results greater than the highest calibration standard must be diluted and reanalyzed, or flagged with an increase measure of uncertainty. The lowest calibration standard must be at or below the Practical Quantitation Limit (LOQ / PQL). Calibration standards must include concentrations at or below the regulatory limit if the laboratory knows these limits, unless these concentrations are below the LOQ / PQL.

Samples and associated quality control samples shall be quantitated from the initial calibration and not a continuing calibration unless the method, regulation, or program requires it.

6.5.2 Continuing Instrument Calibration Verification

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analyses by a continuing instrument calibration verification (CCV) as required. The details of the continuing instrument calibration and acceptance criteria are included in the specific method SOP's. If the CCV fails, it may be re-analyzed. If it fails again, corrective action or a new initial calibration shall be performed. Sample analysis can't begin until a successful CCV or new initial calibration has been performed. Any analysis from an unsuccessful CCV shall be required to be flagged.



When the results for a CCV are high and all associated samples are non-detects, the non-detects may be reported un-flagged. When the results for a CCV are low and all associated samples are non-detects, the non-detects may be reported un-flagged if a standard at the PQL/ LOQ has been analyzed to verify instrument sensitivity.

A CCV must be analyzed at the beginning and end of each analytical batch. If an internal standard is used, only one CCV must be analyzed per analytical batch. Sufficient raw data records must be retained to permit reconstruction of the CCV and must explicitly connect the CCV to the initial calibration.

7.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

7.1 Standard Operating Procedures (SOPs) and Test Methods

GCT follows methods published by international, national, regional, and client supplied standards. Laboratory developed methods shall not be used for compliance requirements. All methods shall be validated prior to use. It shall be the responsibility of the QA Manager and the Director to ensure that the correct procedures are being used. If the correct revision is not in our possession, the client or regulatory agency shall be asked to provide the new revision. The Director or QA Manager shall review and approve new testing procedures to ensure GCT complies with the latest regulations and client needs. All methods or other information pertinent to the analysis, shall be considered controlled documents. The QA Manager will issue unique numbers to the documents to ensure only the most recent version is used. The QA Manager will maintain a master list of the documents. This system ensures that all of the procedures conducted by Green Country Testing are of the latest revision.

Sometimes the EPA or other entity will publish and approve a new revision of a method. The new revision must be evaluated by the Director and QA Manager. It is the intent of the laboratory to use the most recent approved version of a method when possible. Sometimes, two or more revisions of the same method might still be approved. Sometimes, a client's permit states a certain revision of a method to be used. Because of these limiting factors, the laboratory may be forced sometimes to utilize an older revision. If a new method or method revision is approved, it may be necessary for GCT to amend our Scope of Accreditation with various entities. We will request the change to the scope on our annual renewal unless an immediate change is required. If an immediate change is required, we will ask for interim certification.

Standard operating procedures (SOPs) have been developed by Green Country Testing for all procedures and testing performed by GCT staff including referenced methods and laboratory developed methods. Copies of these procedures are controlled by QA. One set of manuals is kept by the QA Manager along with a listing of the location of all controlled copies. Another full set of procedures are located in the laboratory or in the LIMS for easy access by all GCT personnel. All procedures shall be purged from the system when a new revision is put into use. The QA Manager will retain one copy of each outdated revision in a "Superseded" file. For specific and detailed information on standard operating procedures, please refer to the SOP manuals. The listing of the most recent in-house revision of each procedure is on file separate of this manual and is



controlled separately by an approved SOP. Strict document control is exercised over all laboratory manuals and postings.

Any samples that do not fall into the standard SOP's or standard methods shall be dealt with on an individual basis. The analyst shall inform the Laboratory Director or QA/QC Officer and they will come up with a plan on how to handle the unusual sample. This may include the client requesting an out of date method or revision. If the client doesn't specify the method or doesn't know, the laboratory shall select the appropriate published method based on the information available. The client will be informed of the methods selected. Any departures from the SOP or published methods will be documented and the data shall be flagged accordingly.

The SOP's designate sample volumes and/or weights used in the determination. If upon analyst discretion, the normal weight or volume isn't appropriate, (high solids, limited sample, or a smaller volume will still yield reporting limits appropriate to meet regulatory limits) the analyst may choose to utilize a different weight or volume. The actual weight or volume will be recorded in the appropriate logbook and adjustments made to the reporting limit.

For more detailed information, refer to the SOP on the Creation of Revision of SOP's.

7.2 Data Verification

Analytical data generated within Green Country Testing are extensively checked for accuracy and completeness. The data may be additionally verified by inter-laboratory comparison, analysis of PT samples, replicate tests using different methods, retesting of retained samples, or correlation of results for different characteristics. The data validation process usually consists of data generation, reduction, and 3 levels of review, as described below:

Level I

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and produced following protocols specified in referenced methods. Each analyst reviews the quality of his work based on the following set guidelines, where applicable:

- The appropriate methods have been followed with any special sample preparation requirements;
- Sample preparation information is properly documented;
- Analysis information (lab ID, client, test method and result) is correct;
- QC samples are within established control limits;
- Documentation is complete (e.g. all anomalies in the preparation and analysis have been documented, out-of-control rechecks [if required] are complete; holding times are documented, etc.).

The data results are documented, initialed and dated by the analyst. This initial review step, performed by the analyst, is the first level of review. The analyst then passes the



data to an independent reviewer (the Group Leader, Technical Manager, QA Manager, or Director or another qualified analysts trained to run the test being checked) who performs the LEVEL II review.

Level II

A peer, the Technical Manager, QA Manager, or Director performs an independent review of the data. This review is also conducted according to the following set of guidelines and is structured to ensure, where applicable:

- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC check samples are within established guidelines;
- Qualitative identification and quantification of sample components are correct;
- Documentation is complete and correct (e.g. anomalies in the preparation and analysis have been documented; out-of-control rechecks [if required] are complete; holding times are documented, etc.);

Level II reviews are structured so that all calibration data and QC sample results is reviewed. If no problems are found with the data, the review is complete. A date stamp or acknowledgement in the LIMS is used by the reviewer. If any problems are found with the data, all samples in the batch are checked in their entirety. Corrections or amendments to test records are made in a manner that does not obliterate the original data and are dated and initialed by the person responsible.

Level III

The final report is given to the QA Manager, Assistant Director or Environmental Director for final review. The report and data are reviewed to assure that the data package is complete and the results are consistent with method and contract requirements. The final reviewer signs the final report and takes ultimate responsibility for data released to the client.

Rounding Rule: The basic rule used for rounding when reporting data shall be the following: Odd numbers round up and even numbers stay the same.

7.3 Documentation and Labeling of Standards and Reagents

Quality data requires traceable standards of proven purity. To ensure the highest purity possible, all primary reference standards and standard solutions used by Green Country Testing are obtained from the National Bureau of Standards, the EPA Repository or other reliable commercial sources. GCT attempts to use only standards traceable to NIST or traceable back to its original source. Standards are either documented in logbooks or in the LIMS. All records allow for the identification of the standard used and lot number so that it is traceable to the analyses performed. For more information concerning the standards, refer to the standard logs or LIMS.

Stock and working standards are checked with each use for signs of deterioration, such as discoloration, formation of precipitates, or change of concentration. All containers



are properly labeled with unique identifier, compound, concentration, expiration date, initials of analyst, and date of preparation.

Reagents are examined for purity by subjecting a blank aliquot or sub-sample to the analytical methods corresponding to its intended use. All materials shall be stored under appropriate conditions in accordance with OSHA guidelines specific to each chemical to ensure the integrity of the materials and the health and safety of all employees. Analysts shall record lot numbers of all reagents and consumable supplies when available in the appropriate laboratory logbook or within the LIMS system.

7.4 Computers and Electronic Data Related Requirements

All computers and electronic data requirements are developed and maintained by the IS department. For complete details of the program.

For more detailed information, refer to the SOP on Information Technology.

8.0 SAMPLE HANDLING, SAMPLE ACCEPTANCE AND SAMPLE RECEIPT

Data of known quality begins with the collection of the sample. All sampling and ambient data collection is performed in accordance with accepted guidelines and procedures established by the USEPA and Oklahoma Department of Environmental Quality (ODEQ). A number of sampling protocols may be employed to satisfy these project requirements though the ultimate objective for all sampling is to obtain a representative sample. Provisions may be necessary based on specific client requirements in regards to sampling plans. If the client requires deviations, additions, or exclusions from the documented sampling procedure, the details will be recorded and communicated to all the appropriate personnel.

Prior to establishing sampling stations and parameters to be analyzed, a thorough review of program objectives is undertaken by the Account Managers in association with the Director and Field Services group. This review establishes the sampling points, type of sample required, and the analytical parameters to be used in the project. The sampling and parameter information help set the type of field equipment, collection bottles, and preservation chemicals required completing the program.

As part of this review, the client will be an important element for successful completion of the sampling event. Any client may request a copy of our QAM or other SOP's to aid in there ability to ensure that the quality objectives are met. If upon having a conversation with the client we determine that their knowledge may be insufficient or lacking in certain areas, we may choose to send them our QAM or SOP's to further their knowledge on the requirements for sampling and analysis of samples.

Samples must be collected in appropriate containers which have been properly cleaned and to which the correct preservative has been added. This step is important to ensure volatilization, adsorption, or chemical change introduces no contamination. Green Country Testing can assist in the sample collection process with trained sampling technicians and by making available to the client a set of appropriate sample containers that have been properly prepared for use in sample collection.



The maximum holding times recommended by Green Country Testing, appropriate containers and preservatives, and minimum sample volumes required for routine organic, metal and conventional parameters are in agreement with state and federal policies. When the laboratory personnel must split samples, specific standard operating procedures are followed.

8.1 Sample Tracking

The sample is assigned a unique number by the LIMS system. It follows the following parameters: A normal number is 1206111-002A. The first two digits refer to the year received, 2012. The next two digits refer to the month received, June. The next three digits refer to a consecutive number this was the 111^{th} set of samples received in June. The numbers after the dash refer to the number of samples received in this batch from the client, the second sample. The last letter refers to the aliquot of sample (i.e. if a sample was to be analyzed for BOD (unpreserved) and COD (preserved with Sulfuric), there would be two separate containers for the same sample. BOD would be named – 002A and the COD would be –002B). The sample custodian shall print labels for the bottles appropriately.

See the SOP for sample login and receipt for more detailed information.

8.2 Sample Acceptance

All samples must meet the following requirements for acceptance for analysis, where applicable:

- 8.2.1 The chain of custody must be complete and shall include client, sample description, date and time of collection, sampler, preservation type, sample type, relinquishing and received by signatures, and special remarks concerning the sample. Results generated in the field shall be recorded on the COC along with the time of analysis and the analyst initials. The sample custodian shall record the field data in the field data section of the LIMS system.
- 8.2.2 All samples must be properly labeled with a durable label completed in indelible ink and shall include client, description, date and time of collection, sampler, and required tests.
- 8.2.3 All samples shall arrive in proper sample containers with the appropriate preservatives.
- 8.2.4 All samples shall arrive prior to holding time expiration.
- 8.2.5 All samples shall arrive with sufficient sample volume to perform the required tests.
- 8.2.6 All samples that show signs of damage, contamination or inadequate storage shall be brought to the attention of the client.

When any of these criteria are not met, the client shall be notified immediately. If the client insists that the sample be analyzed, the date, time, client's name and specifics of the conversation shall be written in the client conversations logbook or in the LIMS phone log. If the client cannot be contacted, the Laboratory Director, QA Manager, or



Technical Manager shall make the decision for sample acceptance. This information will be documented accordingly.

8.3 Sample Receipt

Samples are received either directly from the client, through a courier such as United Parcel Service or Federal Express or through our field services section (route driver). The samples are checked for label identification and for complete, accurate chain of custody after which the individual performing login signs the chain. The condition of the samples is recorded on the Sample Login Checklist. When questions arise due to the sample's suitability for testing (i.e. discrepancy between ID on sample container and chain of custody or analyses required, etc) the laboratory shall contact the client for further instruction or clarification before proceeding.

Samples follow a two-phase login procedure. This includes assigning a unique identification number to the sample. The second consists of entering the sample login information into a computerized LIMS system, which stores all identification and essential information.

The information recorded within the LIMS system includes the received date, assigned sample number, the client's name and exceptions noted. The sample is checked with pH paper to determine if it is appropriately preserved (where necessary). Any discrepancies are noted on the Chain of Custody. Holding times are verified and the analyst is notified if the holding times are to expire soon. The samples are distributed to the appropriate locations for analysis or to the appropriate storage area until analysis.

8.4 Sample Storage

Samples that are stored under refrigeration require a temperature of 0-6°C (the lower limit is 0°C but not freezing) unless method, specific criteria exists. Separate refrigerators exist for all standards, reagents, food and other potentially contaminating sources. Sample fractions, extracts, leachates and other sample preparation products are stored in the same manner as the original sample.

For more detailed information, refer to the SOP on Sample Receipt and Log-in and also see Appendix IV.

8.5 Sample Disposal

All samples are disposed in compliance with all applicable federal, state, and local guidelines. Samples will be retained for 4 to 8 weeks after sample analysis. Various samples are stored for longer periods at the request of the client.

For more detailed information, refer to the Chemical Hygiene Plan.

9.0 RECORDS



All records that would allow historical reconstruction of laboratory activities are maintained for 7 years. This includes but is not limited to the following:

- 1. Customer purchase orders
- 2. Customer test records and test instructions with data results
- 3. Log in books
- 4. Calibration, standard preparation logs and maintenance logs
- 5. Test reports and chain-of-custody
- 6. Computer database information (transferred and stored on disk and online storage.)
- 7. Any other notes, logbooks, bench sheets, spreadsheets, standard certificates, PT reports, detection limit studies, or other types of records shall be retained

The filing system at Green Country Testing is organized in the following manner: All current information is available online in the LIMS system. Long term storage of data is located in the vault room, which is located in the Organics department or in the sheds outside of the property. As of December 1st, 2011 all versions of reports and revisions are stored digitally within the LIMS.

Only permanent ink and black will be used. No entry will be obliterated by any method. If corrections are to be made, it will be a single line strike through with date and initials of person making the change. The data will be available to any responsible party. In the event of ownership transfer of the laboratory or the laboratory ceases operation, the client shall be notified and the records shall be maintained by the current ownership or transferred to the new ownership or transferred to the client. The laboratory will also comply with any other state or federal requirements concerning record retention.

For more detailed information, refer to the SOP on Sample Receipt and Login

10.0 LABORATORY REPORT FORMAT AND CONTENTS

- 10.1 Reports will contain the following, where applicable:
 - 10.1.1 Cover page with laboratory name and division, client name and address, workorder number (laboratory ID or sample number), approval signature, date sample received, and date of report issuance.
 - 10.1.2 The Case Narrative or comments page will contain the following:
 - a) client generated results such as pH, Dissolved Oxygen, Flows, Temps, Chlorine Residual;
 - b) subcontracted results without existing LIMS test code
 - c) notations of non standard sample conditions such as temperature out of the 0-6°C criteria;
 - d) failed quality control samples that cannot be reanalyzed due to insufficient quantities. failed surrogates will be reported on the analytical report
 - e) deviations from test methods such as emulsions that would require filtration of the sample not called for within the test method;
 - f) and/or notations of missing required items i.e. dates and times of collection.



- 10.1.3 The report also contains the following statements: "This report shall not be reproduced except in full, without the written approval of Green Country Testing, Inc." and "The results relate only to the samples tested."
- 10.1.4 Chain of custody forms or sampling request form with custody information and Sample Login Checklist.
- 10.1.5 The report body shall contain results generated by Green Country Testing employees along with appropriate data flags for failed QC and notes for non-accredited NELAC tests.
- 10.1.6 Results may include any and all QC and related documents associated with sample testing.

When there is exceptionally permitting departures from documented policies and procedures or from standard specifications, the analyst should consult the management of GCT on how to proceed and additionally provide documentation in the appropriate logbook or in the LIMS. Environmental reports are generated by the Laboratory Management System (LIMS).

For more detailed information, refer to the SOP on Sample Receipt and Login.

10.2 Report Revisions and Amendments

Reports may be revised at the request of the customer and approval of the Director. Green Country Testing may also revise the reports upon discovery of an omission or error. Pertinent information is documented in the case narrative section of Omega and in the amended report logbook located in the Customer Service department. The original report is kept unchanged and a new report is generated within the LIMS as necessary.

10.3 Faxing/Mailing Reports

Reports will be faxed to customers upon their request. Reports may also be faxed at the request of the Director, QC staff, or Administrative Assistant's discretion.

Faxed transmissions will include the following:

"<u>Confidentiality Notice</u>: This facsimile transmission (and/or the documents accompanying it) may contain confidential information belonging to the sender. The information is intended only for the use of the individual or entity named above. If you are not the intended recipient, you are hereby notified that any disclosure, copying, distribution or the taking of any action in reliance on the contents of this transmission is strictly prohibited. If you have received this transmission in error, please immediately notify us by telephone to arrange for the return of the documents."

Reports will only be mailed, faxed or emailed to those persons and companies whom have been specifically designated by the customer to receive them. Clients are notified via Customer Service within 48 hours or two business days when events cast doubt on the validity of test results.

11.0 SUBCONTRACTING ANALYTICAL SAMPLES

Only certified companies will perform calibrations of laboratory equipment. These certifications are kept on file in the QA files. Analytical services not performed by



Green Country Testing will ONLY be sent to certified laboratories as required. Copies of these certifications are kept on in the QA files. Clients are notified in writing of subcontracted work and the notification and approval, if available, will be maintained in the LIMS. Lab reports denote subcontracted work with their certification number in the analyst field.

Appropriate chain-of-custody procedures are followed when submitting samples to a subcontractor for analysis. Subcontracted reports are kept with the login paperwork in the designated file cabinet or scanned into the associated work order within the LIMS system. All subcontractors must show conformance to Green Country Testing quality requirements. Where available, standard operating procedures will be requested and filed in the QA files.

12.0 OUTSIDE SUPPORT SERVICES AND SUPPLIES

Outside support and supplies will be purchased from sources qualified and/or approved by the Director and/or QA Manager. The laboratory will purchase only those supplies that are of adequate quality to provide confidence regarding the reliability of the analysis. All supplies and equipment purchased will be inspected and tested (where appropriate) to verify the quality of the item. Under no circumstances will consumable supplies be used until inspection is complete.

The purchasing agent will maintain records of all suppliers from who support services or supplies are purchased. The purchasing agent may choose a vendor when negotiating a lower price as long as the quality of the item is equivalent to or better than the original. The Director must give approval for the changes. Copies of purchase orders, packing slips, and vendor invoices are maintained in a separate file.

For more detailed information, refer to the SOP on Reception, Storage and Labeling of Consumables

13.0 COMPLAINTS

Complaints by clients or personnel for any circumstances raising doubt about the laboratory's adherence to policy or procedure will require an internal audit for resolution. Once resolved, full documentation of the corrective action procedure is filed for future reference. The QAO shall be informed of situations that may lead to Corrective Actions and documented in the Customer Complaint logbook appropriately. The QA Manager and/or Director routinely review corrective action documentation. For other complaints or comments from clients, a logbook is kept by Customer Service to document these occurrences so that we might determine trends or other courses of action.

14.0 ETHICS AND DISCIPLINARY POLICY

14.1 Ethics Policy

The successful business operation and reputation of Green Country Testing is built upon the principles of fair dealing and ethical conduct of our employees. Our reputation for



integrity and excellence requires careful observance of the spirit and letter of all applicable laws and regulations, as well as a scrupulous regard for the highest standards of conduct and personal integrity.

The continued success of Green Country Testing is dependent upon our customers' trust, and we are dedicated to preserving that trust. Employees owe a duty to Green Country Testing, its customers, and shareholders to act in a way that will merit the continued trust and confidence of the public.

GCT will comply with all applicable laws and regulations and expects employees to conduct business in accordance the letter and spirit of all applicable laws and to refrain from any illegal, dishonest, or unethical conduct.

In general, the use of good judgement, based on high ethical principles, will guide you with respect to acceptable conduct. If a situation arises where it is difficult to determine the proper course of action, the matter shall be discussed openly with the Laboratory Director and Quality Assurance Officer.

Compliance with this policy of ethics is the responsibility of every employee. Disregarding or failing to comply with this policy could lead to disciplinary action, up to and including possible termination of employment.

Ethics training will be conducted on an annually basis by the Quality Assurance Officer. Annual ethics and data integrity procedures shall be reviewed and training shall include documentation that data integrity practices, such as breaches of ethical behavior and the consequences for not complying with company policy have been communicated and are understood by each employee.

For more detailed information, refer to the SOP on Employee Training

14.2 Disciplinary Policy

This policy states GCT's position on administering equitable and consistent discipline for deviations from the policies within this manual. The major purpose of any disciplinary action is to correct the problem, prevent recurrence, and prepare the employee for satisfactory service in the future.

Disciplinary action may call for any of four measures – verbal warning, written warning, suspension with or without pay, or termination of employment- depending on the severity of the problem and the number of occurrences. There may be circumstances when the measures are followed in progression. Use of discipline is intended to correct problems at an early stage, benefiting both the employee and Green Country Testing.

15.0 MANUALS, LOGBOOKS, AND FORMS REGISTRATION AND DISTRIBUTION

The quality manual and laboratory logbooks shall be distributed and maintained on a controlled copy basis. Manuals released for distribution must be signed by authorized personnel. All Management System Documents including registered manuals and



logbooks must be accounted for on the internal document control log located in the QA/QC office. Each time a manual or logbook is distributed, it is given a unique ID number in either the Quality Manual log or the laboratory notebook log with the start date and GCT ID number on the front cover. It is the responsibility of each employee who receives a registered manual to read and understand the contents of the manual. The QAM and other laboratory documents such as the Employee Handbook, the Chemical Hygiene Plan and Safety Manual can be accessed from the LIMS system under Documents and SOP's.

The quality manual shall be reviewed annually to ensure continued correctness and applicability. When required, interim reviews shall be made. Notification of proposed additions and/or amendments may be made by anyone at any time and submitted to the Quality Assurance Manager. All requests shall be reviewed by the Director and QA Manager for approval. Approved additions and/or amendments shall be distributed to registered manual holders. It is the responsibility of each registered manual holder to enter the approved amendment in his or her manual. Amendments within each manual revision shall be numbered consecutively and records maintained on the list of approved revisions/amendments. All users have read only access to sensitive documents, however management and QA personnel at GCT have full control to make the necessary changes to these documents. All external documents shall be given a unique ID number in the external document control log located in the QA/QC office.

Various forms and other information may be used within the laboratory. All forms and other information will be given a unique identifier by the QA Manger and tracked accordingly. Forms may include refrigerator monitor records, min/max records, or other forms used within the laboratory. Other information may include regulations, methods, equipment manuals, signature records, facility maps, evacuation maps, fire extinguisher locations, or other information.



APPENDIX I - GLOSSARY

Accuracy Acid digestion	The ability of a test to give the true amount of target analyte. Method for obtaining metal analytes in solution for analysis.	
Acute toxicity	Short-term lethal effects, generally 4 days or less for fish and 2 days form smaller organisms such as microcrustacea.	
Alkalinity	A measure of the acid-neutralizing ability of the sample.	
Analytical balance	Electronic balance capable of accurate weighings to 0.1 mg.	
ASTM	American Society for Testing Materials.	
Atomic Absorption (AA)	Method of analysis based on atomizing a metal sample in a flame and	
	monitoring the absorption of specific wavelengths of light passed through the	
	flame.	
Audits	Examination of test procedures or other laboratory processes and comparison of	
	the findings to a written standard; may be performed by an internal or external	
	person to the laboratory.	
Base-Neutral Acids (BNA)	Compounds extractable from basic, neutral, or water solutions with organic	
	solvents.	
Base-Neutral Extractables	Compounds extractable from basic or neutral water solutions with organic	
	solvents.	
BFB Tuning Requirements	Abundance and mass detection requirements that must be met before analysis of	
Di la	compounds begins.	
Bioassay	A technique for determining the power of a substance by measuring its effect on	
Rischamical Ormon Domond (D	a test specimen against those of a standard substance.	
Biochemical Oxygen Demand (B		
BTEX Buffers	Benzene, Toluene, Ethylbenzene, and total Xylenes test. Solutions of a weak acid and a salt of the acid or a weak base and a salt of the	
Builers	base that are capable of maintaining pH on addition of acid or base.	
Calibration curve	Graphical plot of instrument response against amount of analyte in standards.	
Calibration curve	Most often gives a linear (straight line) result.	
California Method	Slang term for the GC FID determination of diesel fuel.	
CBOD	Carbonaceous Biochemical Oxygen Demand.	
CFR	Code of Federal Regulations.	
Chain of Custody	Legal document recording who had the sample and over what period of time as	
	it moves from the sampling point to the laboratory.	
Chlorination	Addition of chlorine to water for disinfection purposes.	
Chronic toxicity	Longer term lethal or reproductive effects than acute toxicity. The response to a	
	particular concentration of a toxicant will vary from species to species.	
	Concentrations for chronic effects may be found at as little as 10 ⁻⁶ times the	
	concentration that results in acute effects. Although some true chronic toxicity	
	studies are performed, extending over periods of months in the environmental	
	industry, short-term methods are used that serve to estimate long term effects.	
CLP	Contract Laboratory Program.	
CLP Deliverables	Required quality control results and analytical documentation that must	
	accompany each sample report on submission to the EPA under the CLP.	
COD California ha dania	Chemical Oxygen Demand.	
Coliform bacteria	Any anaerobic and aerobic, gram negative, non-spore forming, rod-shaped	
	bacteria that ferment lactose within 48 hours at $35\Box C$ to produce carbon dioxide and acid.	
Composite semples		
Composite samples	Individual samples are mixed together to make a single sample. tion (CCV) Standards are reanalyzed on a daily basis to reconfirm the	
Continuing Calibration Verification (CCV) Standards are reanalyzed on a daily basis to reconfirm the validity of the instrument calibration.		
Control charts	Day-by-day or batch-by-batch plots of accuracy or precision as a process	
Control chur to	monitor.	



Conventional pollutants	BOD ₅ , TSS, pH, and Fecal Coliform tests standard on most NPDES permits.
Correlation Coefficient	A mathematical measure of the goodness of fit, ranges from 0 to 1 with 1 being
	a perfect fit.
Definitive test	Multiple replicates of at least five different concentrations of test material, the
	concentrations must bracket the instream water concentration and the end-point
	concentration.
Diesel Range Organics (DRO)	Organic compounds determined by GC FID that has retention times between
	decane and pentacosane, often assumed as diesel fuel.
Digestion	Solubilizing of metal analytes through heating with a variety of acids or
8	oxidizers.
Discharge Monitoring Report (I	
	required of NPDES permit holders.
EPA	Environmental Protection Agency.
End-point	The observable effect of the test generally limited to death, growth impairment,
Lind point	and/or reproductive success.
Equipment Blanks	Sampling equipment is rinsed with reagent water that is tested to determine
Equipment Dums	contamination levels on the equipment.
Fecal Coliform	Coliform bacteria found in mammalian intestinal tracts.
FID	Flame Ionization Detector.
Flash Point (Ignitability)	Temperature at which a sample creates sufficient vapor to support combustion in
Flash Font (Ignitability)	air.
Gas Chromatography (GC)	Separatory technique that uses a solid or liquid absorbent and an inert gas stream
Gas Chromatography (GC)	as mobile phase – separation mechanisms are vapor pressure and chemical
	affinity.
Casalina Panga Organias (CPO) All organic materials that elute from a GC with retention times between 2-
Gasonne Kange Organics (GKO	methyl pentane and trimethylbenzene – assumed to be gasoline fuel.
GCMS	Gas Chromatography – mass spectrometer interfaced together.
Grab sample	A sample obtained in a single instant of time at a single location.
Graphite Furnace	A carbon tube that is electrically heated in an AA to replace the flame as the
Graphite Furnace	atomization source.
Groundwater	Subsurface water.
Hardness	Dissolved metal content of water, expressed as calcium carbonate.
High Performance Liquid Chro	
Ingh i eriormance Eiquiu Chro	liquid mobile phase – separation mechanism is chemical partitioning.
Holding times	Elapsed time between moment of sampling and initiation of analysis.
Hotplate digestion	Open beaker method of acid digestion of metals.
ICAL	Initial calibration.
Indicators	Chemicals added to a sample that signal an end to a process by a color change.
Inductively Coupled Plasma (IC	
inductively Coupled I fashia (IC	metal atoms in emission spectrometry.
Internal standards	Compounds added to the sample after sample preparation for qualitative and
Internal standards	quantitative instrument analysis- the compounds serve to give a standard of
	retention time and response, which is invariant from run to run with the
	instruments.
Ion Chromatography (IC)	Instrument very similar to an HPLC used for analysis of anions or cations.
Laboratory Control Sample (LC)	
Laboratory Control Sample (LC	analyzed. Serves as a quality control on the sample preparation process.
LC50	Lethal concentration for 50% of the test subjects.
	Laboratory Control Sample.
Legally defensible	
Legany uciensible	Analytical results supported by the necessary documentation to withstand
Lathal concentration I ()	challenge in a US court of law.
Lethal concentration LC)	The toxicant concentration that kills a specified percentage of test organisms after a set observation time, expressed as 48 h I Cr., where a 48 hour test was
	after a set observation time, expressed as 48 -h LC ₅₀ , where a 48 -hour test was
	performed and 50% of the organisms died.



LFB	Laboratory Fortified Blank
LIMS	Laboratory Information Management System.
LOEC	Lowest Observable Effect Concentration. The lowest concentration of test
	substance that produces an effect. The lowest concentration that produces a
	statistically significant difference between the controls and the test samples.
M-FC	Tradename for a microbiological media.
Matrix Spike (MS)	Target analyte added to a sample in known amount to determine recovery for the
	specific matrix.
Maximum Contaminant Level (
Method Detection Limit (MDL)	
MSDS	Material Safety Data Sheet.
NELAC	National Environmental Laboratory Accreditation Committee.
NELAP	National Environmental Laboratory Accreditation Program.
NIST	National Institute for Standard and Technology (formerly NBS).
Nitrogen inhibition	Chemicals added to CBOD test to eliminate nitrogen oxidation demand on the
0	results.
NOEC	No observable effect concentration. The maximum concentration of test
	substance that produces no effect. No effect is designed as any statistically
	significant difference between the controls and the test samples.
NPDES	National Pollutant Discharge Elimination System.
NTU	Nephelometric Turbidity Unit.
РСВ	Polychlorinated biphenyl.
pH	The negative log of the hydrogen ion expressed as standard units (S.U.).
PID	Photoionization Detector.
POTW	Publicly Owned Treatment Works.
ppm	Part per million.
Precision	The closeness of agreement between two or more analyses of the same sample.
Proficiency Test Samples (PT)	Blind sample sent to the laboratory by outside parties for evaluation of
	qualitative and quantitative accuracy.
Preservatives	Chemical or physical treatment of the sample to assure of the same sample.
Qualified data	Data derived from a sample analysis where something about the sample, the
	sampling procedure, or the analysis is not in accordance with specifications.
Qualitative analysis	The analytical process to determine what is in the sample.
Quality Assurance Program	The written overall program in the laboratory that details all the steps and
	procedures used to produce data of a known and stated quality.
Quality Control (QC)	An individual procedure used in the quality assurance program. A matrix spike
	performed on a metals sample is a quality control, the use of matrix spikes in all
	procedures in the laboratory is quality assurance.
Quantitative analysis	After an analyte is identified in the sample, quantitative analysis is used to
	determine how much is there.
Range Finder (Screening Test)	An acute test or short-term chronic test performed with a single replicate at
	multiple concentrations of test material.
Reference Toxicant	A standard material (potassium chloride, sodium dodecylsulfate, potassium
	dichromate, cadmium chloride, sodium chloride, etc.) that has a well-
	characterized quantitative dose-effect on the test organism. Used as a quality
	control in toxicity tests.
Relative Percent Difference	The difference between two values divided average of the values, expressed as a
Donnocontativo comula	percent. The degree to which a single comple of the whole congive results identical to
Representative sample	The degree to which a single sample of the whole can give results identical to
Dognongo Footor (DE)	analysis of the whole. Multiplication factors for converting instrument response to mass of analyte
Response Factor (RF) Retention time	Multiplication factors for converting instrument response to mass of analyte.
Netention time	Elapsed time between injection of sample to elution on the chromatogram.



SMC

Static non-renewal test

Static renewal test

Surrogates

TAT TCLP Toxicant

Trip Blanks

VOA VOC's Whole Effluent Toxicity (WET) System Monitoring Compounds (Surrogates), compounds added to the sample prior to preparation, then monitored after analysis to detect problems in the sample preparation procedure.

The test organism is exposed to a single portion of the test solution for the duration of the test. The test is limited due to the shortage of dissolved oxygen and nutrients in the single portion of solution and the build-up of test organism waste products.

The test organism is exposed to fresh changes of the test material in water every day for the duration of the test.

Compounds added to the sample prior to preparation, then monitored after analysis to detect problems in the sample preparation procedure.

Turn-around time.

Toxicity Characteristic Leaching Procedure.

A substance that is harmful to living organisms due to detrimental effects on tissue, organs, or biological processes.

VOC sample containers assembled with reagent water at the laboratory, then taken to the field and back to the lab, analyzed to reveal contamination that occurs by passage of volatile compounds through the septum into the containers. Volatile Organic Analysis.

Volatile Organic Compounds. A technique for determining the power

A technique for determining the power of an effluent by measuring its effect on a test specimen against those of a standard substance or control.



APPENDIX II - PERSONNEL

Brian Duzan, Director of Environmental Services

B.A. Chemistry, 1989, Blackburn College - Carlinville, Illinois

<u>Applied Research and Development Laboratory</u>, Mt. Vernon, IL – (1989-1990): Employed as a chemist in the metals department.

Southwest Labs of Oklahoma, Broken Arrow, OK – (1990-1991): Employed as a chemist in the metals department.

<u>T.I. Laboratories</u>, Tulsa, OK – (1991-1995): Employed as metals chemist, organics chemist, then Laboratory Director.

<u>Outreach Laboratories</u>, Broken Arrow, OK (1995-1999): Employed as QA/QC Officer and then Laboratory Manager.

<u>Green Country Testing</u>, formerly Sherry Laboratories/Oklahoma (since 1999): Responsible for day-to-day staff management including hiring, performance assessment and safety program. Additional responsibilities include assuring adherence to EPA and other regulatory methods, monitor performance on external as well as in-house analytical reference samples to maintain regulatory certification and assure on-going quality of data generated. Responsible for purchasing and assisting the president with preparation and monitoring of operating and capital budgets. Responsible for organics analysis by GCMS. Special training courses include: Hewlett Packard GC Maintenance Operation Course.

Star Yuan, Environmental QA Manager

B.A. Environmental Policy, 1999, University of Tulsa – Tulsa, Oklahoma

<u>Green Country Testing</u>, formerly Sherry Laboratories/Oklahoma (since 1999): Responsible for the day to day implementation of the Quality Program. Special training courses include HazWop Training (40 hr course in 1999).

Craig Walker, Laboratory Analyst

B.S. Environmental Biology, 2015, Rogers State University

<u>Ana-Lab Corporation</u>, Broken Arrow, OK, (2015-2016) –Responsible for performing field testing on water and soil samples, as well as collecting specimens to be sent for more extensive testing.

<u>Sherry Laboratories</u>-Broken Arrow, OK, (2008-2012)-In the Non-Metallics division, reading, interpreting and writing up plans for test specifications. Also performed routine testing for aerospace companies, drafting court reports for testing done, and various customer service responsibilities.

<u>Green Country Testing</u>, full time since 2016: Primarily responsible for Oil and Grease testing and various wet chemistry analysis.

Keli Pilkington, Chromatography Analyst

B.S. Biology, 2003, Oklahoma State University – Stillwater, Oklahoma

<u>Green Country Testing</u>, formerly Sherry Laboratories/Oklahoma full time since 2003: Primarily responsible for preparing and analyzing samples on the Lachat autoanalyzer and other wet chemistry procedures. Performs chromatography procedures in the organics department. Heads the IT department and is responsible for all computer related issues.

Currently identified as technical director for microbiological analysis.

Bryan Grey, Laboratory Analyst

<u>City of Tulsa</u>, Tulsa, OK, (2018–2019) – Performed wet chemistry procedures. <u>Green Country Testing</u>, full time since 2018: Primarily responsible for wet chemistry testing.



Dakota Wilkinson, Laboratory Analyst

B.A. Psychology Minor Health and Exercise, Oklahoma University - Norman, Oklahoma

Pace Analytical, Tulsa, OK, (1996–2007) – Performed BOD, CBOD, Micro, and many wet chemistry procedures.

Green Country Testing, full time since 2019: Primarily responsible for wet chemistry testing.

Kristi Ryan, Laboratory Analyst

<u>USPCI/Laidlaw/NAL</u> (1995 to 1997): Preparation of samples for metals analysis, preparation of TCLP extracts.

<u>Green Country Testing</u>, formerly Sherry Laboratories/Oklahoma (since 1997): Instrumental analysis by ICP, TOC and spectrophotometric instruments. Wet methods include gravimetric and titrimetric techniques. Special training includes: HazWOp training (24 hr course, 1997). Member American Chemical Society.

Janet Fitzgerald, Support Services Coordinator & Sales Manager

<u>Southwest Laboratories</u> (1998-2001): Lab testing including: TCLP testing, TOX, and TOC. <u>Green Country Testing</u>, formerly Sherry Laboratories/Oklahoma since 2001: Primarily responsible for the coordination between clients and laboratory in addition to various sales duties.

Keri Little, Support Services

Business Management, University of Phoenix – Tulsa, Oklahoma

<u>Green Country Testing</u>, full time since 2019: Primarily responsible for the log-in of samples and customer service.

<u>Linde Process Plants</u> (2008-2011): SHEQ Coordinator <u>TCI Services</u> (2017-2018): Administrative Assistant <u>Shamrock Industries</u> (2018-2019): QA/HSE Coordinator

Matt Hagan, Lab / Field Technician

<u>Green Country Testing</u>, Green Country Testing employee full time since 2019. Primarily responsible for field work and some laboratory testing. Accurate Testing, (2016-2019) Primarily responsible for field work and some laboratory testing.

Wendell Anderson, Field Technician

<u>Green Country Testing</u>, Green Country Testing employee part time since 2014. Primarily responsible for field work.

<u>Mid-America Industrial Lab</u> (2008-2014): Primarily responsible for field work and some basic laboratory testing.

Matt Brooks, Field Technician

Green Country Testing: Green Country Testing employee since 2019. Primarily responsible for field work.



APPENDIX III – TEST METHOD REFERENCES

"Test Methods for Evaluating Solid Waste, Physical and Chemical Methods," SW-846 [Third edition (November 1986), as amended by Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), and III (December 1996)]

"Methods for Chemical Analysis of Water and Wastes" EPA 600/4-79-020 (Revised March 1983 and 1979 where applicable)

"Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater" Test Methods and Summaries (EPA 600/4-82/057)

"Microbiological Methods for Monitoring the Environment" (EPA 600/8-78-017)

ODEQ methods, (GRO/DRO), for analyzing underground storage tanks (UST).

"Standard Methods for the Examination of Water and Wastewater"-20th Edition (American Public Health Administration).

"Standard Methods for the Examination of Water and Wastewater"-22nd Edition (American Public Health Administration).





APPENDIX IV REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR AQUEOUS SAMPLES

Parameter	Container ¹	Minimum Sample Size, mL	Preservation ^{2,3}	Max. Holding Time ⁴
Bacterial Tests:		IIIL		
Coliform, fecal and total	P,G	200	Cool, $\leq 6^{\circ}$ C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ¹⁵
Fecal streptococci	P,G	200	$Cool, < 6^{\circ}C, 0.008\% Na_2S_2O_3^{-5}$	8 hours ¹⁵
Enterococci	P,G	200	Cool, $\leq 6^{\circ}$ C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ¹⁵
E. coli	P,G	200	Cool, $\leq 6^{\circ}$ C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ¹⁵
norganic Tests:	1,0	200	$\underline{<00}, \underline{<0}, \underline{<0}, 0.00070, 140_{2}0_{2}0_{3}$	0 Hours
Acidity	P,G	100	$Cool, \leq 6^{\circ}C$	14 days
Alkalinity	P,G	200	$Cool, \leq 6^{\circ}C$	14 days
Ammonia	P,G	500	Cool, $\leq 6^{\circ}$ C, H ₂ SO ₄ to pH <2	28 days
Biochemical oxygen demand	P,G	1000	$Cool, \leq 0 C, \Pi_2 SO_4 to p\Pi < 2$ $Cool, < 4^{\circ}C$	48 hours
Bromide	P,G		None required.	28 days
Biochemical oxygen demand,	P,G	1000	Cool, $< 4^{\circ}$ C	
carbonaceous				48 hours
Chemical oxygen demand	P,G	100	Cool, $\leq 6^{\circ}$ C, H ₂ SO ₄ to pH <2	28 days
Chloride	P,G	100	None required.	28 days
Chlorine, total residual	P,G	500	None required.	Analyze immediately
Color	P,G	500	$\text{Cool}, \leq 6^{\circ}\text{C}$	48 hours
Cyanide, total and amenable to chlorination	P,G	500, 500	Cool, \leq 6°C, NaOH to pH >12, 0.6g ascorbic acid	14 days ⁶
Fluoride	Р	300	None required.	28 days
Hardness	P,G	100	HNO ₃ or H ₂ SO ₄ to pH <2	6 months
Hydrogen ion (ph)	P,G		None required.	Analyze immediately
Kjeldahl and organic nitrogen Metals ⁷ :	P,G	500	Cool, $\leq 6^{\circ}$ C, H ₂ SO ₄ to pH <2	28 days
Boron	P(PTFE), Quartz	100	HNO ₃ to pH <2	6 months
Chromium VI	P,G	300	$Cool, \leq 6^{\circ}C$	24 hours
Mercury	P,G	500	HNO_3 to pH <2	28 days
All other metals	P,G	100	HNO ₃ to pH <2	6 months
Nitrate	P,G	100	$Cool, < 6^{\circ}C$	48 hours
Nitrate-nitrite	P,G	100	Cool, $\leq 6^{\circ}$ C, H ₂ SO ₄ to pH <2	28 days
Nitrite	P,G	100	$Cool, \leq 6^{\circ}C$	48 hours
Oil and grease	G G	1000	Cool, $\leq 6^{\circ}$ C, H ₂ SO ₄ or HCl to	
On and grease	0	1000	$c_{001}, \leq 0 C, H_2 SO_4 \text{ of HCI to}$ pH <2	28 days
Organic carbon	G	50	Cool, \leq 6°C, H ₂ SO ₄ or HCl or H ₃ PO ₄	28 days
			to pH <2	
Orthophosphate	P,G	100	Filter immediately, $cool, \leq 6^{\circ}C$	48 hours
Oxygen, dissolved (probe)	G bottle & top		None required	Analyze immediately
Oxygen, dissolved (Winkler)	G bottle & top		Fix on site and store in dark	8 hours
Phenols	G only	500	Cool, \leq 6°C, H ₂ SO ₄ to pH <2	28 days
Phosphorus (elemental)	G	100	$Cool, \leq 6^{\circ}C$	48 hours
Phosphorus, total	P, G	100	Cool, $\leq 6^{\circ}$ C, H ₂ SO ₄ to pH <2	28 days
Residue, total	P,G	100	Cool, $\leq 6^{\circ}$ C	7 days
Residue, filterable	P,G	100	$Cool, \leq 6^{\circ}C$	7 days
Residue, nonfilterable (TSS)	P,G	100	$Cool, \leq 6^{\circ}C$	7 days
Residue, settleable	P,G	1000	$Cool, \leq 6^{\circ}C$	48 hours
Residue, volatile	P,G	100	$Cool, \leq 6^{\circ}C$	7 days
Silica	P(PTFE), Quartz	100	$\operatorname{Cool}, \leq 6^{\circ}\mathrm{C}$	28 days
Specific conductance	P,G	500	$Cool, < 6^{\circ}C$	28 days
Sulfate	P,G	100	$Cool, < 6^{\circ}C$	28 days



REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR AQUEOUS SAMPLES

	Container ¹	Minimum Sample Size, mL	Preservation ^{2:3}	Max. Holding Time ⁴
Sulfide		100	Cool, ≤ 6°C, add zinc acetate plus NaOH to pH >9	7 days
Sulfite	P,G	100	None required.	Analyze immediately
Surfactants	P,G	1000	$Cool, \leq 6^{\circ}C$	48 hours
Temperature	P,G		None required	Analyze immediately
Turbidity	P,G	100	$Cool, < 6^{\circ}C$	48 hours
Organic Tests ⁸				
Purgeable halocarbons	G, Teflon-lined septum	2 x 40	Cool, $\leq 6^{\circ}$ C 0.008% Na ₂ S ₂ O ₃ ⁵	14 days
Purgeable aromatic hydrocarbons	G, Teflon-lined septum	2 x 40	Cool, $\leq 6^{\circ}$ C, 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl	14 days
			to pH 2 ⁹	
Acrolein & acrylonitrile	G, Teflon-lined septum	2 x 40	Cool, $\leq 6^{\circ}$ C, 0.008% Na _s S ₂ O ₃ ⁵ , adjust pH to 4-5 ¹⁰	14 days
Phenols ¹¹	G, Teflon-lined cap	1000	Cool, $\leq 6^{\circ}$ C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
Benzidines ¹¹	G, Teflon-lined cap	1000	Cool, $\leq 6^{\circ}$ C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction ¹³
Phthalate esters ¹¹	G, Teflon-lined cap	1000	$\operatorname{Cool}, \leq 6^{\circ} \mathrm{C}$	7 days until extraction, 40 days after extraction
Nitrosamines ^{11,14}	G, Teflon-lined cap	1000	Cool, \leq 6°C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
PCBs ¹¹ acrylonitrile	G, Teflon-lined cap	1000	$\operatorname{Cool}, \leq 6^{\circ}\mathrm{C}$	7 days until extraction, 40 days after extraction
Nitroaromatics & isophorone ¹¹	G, Teflon-lined cap	1000	Cool, \leq 6°C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons ¹¹	G, Teflon-lined cap	1000	Cool, $\leq 6^{\circ}$ C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
Haloethers ¹¹	G, Teflon-lined cap	1000	$\text{Cool} \leq 6^{\circ}\text{C}, 0.008\% \text{ Na}_2\text{S}_2\text{O}_3^5$	7 days until extraction, 40 days after extraction
Chlorinated hydrocarbons ¹¹	G, Teflon-lined cap	1000	$Cool, \leq 6^{\circ}C$	7 days until extraction, 40 days after extraction
TCDD ¹¹	G, Teflon-lined	1000	Cool, $\leq 6^{\circ}$ C, 0.008% Na _s S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
	G, Teflon-lined	1000	Cool, $\leq 6^{\circ}$ C, pH 5-9 ¹⁵	7 days until extraction, 40 days after extraction



REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR SOILS/SOLIDS

Parameter	Container	Minimum Sample Size, g	Preservation	Max. Holding Time
norganic Tests:		8		
Ammonia	P,G	50	$Cool, \leq 6^{\circ}C$	28 days
Bromide	P,G	50	None required.	28 days
Chloride	P,G	50	None required.	28 days
Cyanide, total and amenable to	P,G	10, 10	Cool, $< 6^{\circ}$ C	14 days
chlorination				
Fluoride	Р	50	None required.	28 days
Hydrogen ion (ph)	P,G	50	None required.	Analyze immediately
Kjeldahl and organic nitrogen	P,G	50	Cool, $\leq 6^{\circ}$ C	28 days
Metals:				
Boron	P(PTFE), Quartz	10	$Cool, \leq 6^{\circ}C$	6 months
Mercury	P.G	10	$Cool, < 6^{\circ}C$	28 days
All other metals	P.G	10	$Cool, < 6^{\circ}C$	6 months
Nitrate	P,G	50	$Cool, < 6^{\circ}C$	48 hours
Nitrate-nitrite	P,G	50		28 days
	P,G	50 50	$Cool, \leq 6^{\circ}C$	48 hours
Nitrite			$Cool, \leq 6^{\circ}C$	
Oil and grease	G	50	$Cool, \leq 6^{\circ}C$	28 days
Organic carbon	G	50	$Cool, \leq 6^{\circ}C$	28 days
Orthophosphate	P,G	50	$Cool, \leq 6^{\circ}C$	48 hours
Phenols	G only	10	$Cool, \leq 6^{\circ}C$	28 days
Dhoomhomic (alamantal)	G	10	Coal < 60C	48 hours
Phosphorus (elemental)			$Cool, \leq 6^{\circ}C$	
Phosphorus, total	P, G	10	$Cool, \leq 6^{\circ}C$	28 days
Specific conductance	P,G	50	$Cool, \leq 6^{\circ}C$	28 days
Sulfate	P,G	50	$Cool, \leq 6^{\circ}C$	28 days
Sulfide	P,G	50	$Cool, \leq 6^{\circ}C$	7 days
Sulfite	P,G	50	None required.	Analyze immediately
Organic Tests				
Purgeable halocarbons	G, Teflon-lined septum	100	Cool, <u><</u> 6°C	14 days
Purgeable aromatic	G, Teflon-lined septum	100	$Cool, \leq 6^{\circ}C$	14 days
hydrocarbons	i i i i i i i i i i i i i i i i i i i			
Acrolein & acrylonitrile	G, Teflon-lined septum	100	$Cool, < 6^{\circ}C$	14 days
Phenols	G, Teflon-lined cap	100	$Cool, \leq 6^{\circ}C$	7 days until extraction,
Filehois	O, Terion-Inied Cap	100	C001, <u><</u> 0 C	
D 11		100	G 1 (0G	40 days after extraction
Benzidines	G, Teflon-lined cap	100	$Cool, \leq 6^{\circ}C$	7 days until extraction
Phthalate esters	G, Teflon-lined cap	100	Cool, $\leq 6^{\circ}$ C	7 days until extraction,
		100 A 100 A		40 days after extraction
Nitrosamines	G, Teflon-lined cap	100	$Cool, \leq 6^{\circ}C$	7 days until extraction,
				40 days after extraction
PCBs acrylonitrile	G, Teflon-lined cap	100	$Cool, \leq 6^{\circ}C$	7 days until extraction,
				40 days after extraction
Nitroaromatics & isophorone	G, Teflon-lined cap	100	$Cool, < 6^{\circ}C$	7 days until extraction,
- in our official of a hopficione	o, renon inter cup	100		40 days after extraction
Polynuclear aromatic	G, Teflon-lined cap	100	Cool, $\leq 6^{\circ}$ C	7 days until extraction,
	o, renon-inted cap	100	$COOI, \leq 0 C$	
hydrocarbons		100	0 1 . 000	40 days after extraction
Haloethers	G, Teflon-lined cap	100	$Cool, \leq 6^{\circ}C$	7 days until extraction,
				40 days after extraction
Chlorinated hydrocarbons	G, Teflon-lined cap	100	Cool, $\leq 6^{\circ}C$	7 days until extraction,
				40 days after extraction
TCDD	G, Teflon-lined cap	100	$Cool, \leq 6^{\circ}C$	7 days until extraction,
	-, · · · · · · · · · · · · · · · · · · ·			40 days after extraction
Pesticides	G, Teflon-lined cap	100	$Cool, \leq 6^{\circ}C$	7 days until extraction,
i concluco	o, renon-meu cap	100	$\underline{c}_{001}, \underline{<}_{0} \underline{c}_{0}$	40 days after extraction



Table Notes

- ¹ Polyethylene (P) or Glass (G)
 - 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 Mail, 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 such compliance. 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); Sulfuric acid (HzSO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.62 or greater); Sulfuric acid (HaCH) in water solutions at concentrations of 0.35% by weight or less (pH about 1.2.30 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. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under §136.3(e). Some samples may not be stable for the maximum time period given. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See §136.3(e) for details. The term "analyze immediately" usually means within 15 minutes or less of sample collection.
- ⁵ Should only be used in the presence of residual chlorine.
- ⁶ Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered, and then NaOH is added to pH 12.
- ⁷ Samples should be filtered immediately on-site before adding preservative for dissolved metals.
 Suidance applies to samples to be applyzed by CC_LC_er CC/MS for specific compounds.
- ⁸ Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds. Sample requiring no pH adjustment must be applyzed within source days of compling.
- ⁹ Sample receiving no pH adjustment must be analyzed within seven days of sampling. ¹⁰ The pH adjustment is not required if agregin will not be measured. Samples for agreging the seven days of samples for a
- ¹⁰ The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- ¹¹ 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 chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for seven days before extraction and for forty days after extraction.
- ¹² If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 \pm 0.2 to prevent rearrangement to benzidine.
- ¹³ Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- ¹⁴ For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- ¹⁵ Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.



APPENDIX V

SOME OF THE METHODS ACCREDITED FOR BY VARIOUS STATES

TEST	WATER METHOD	SOIL METHOD
Bacterial Tests		
Coliform, fecal	9222D, 1997	N/A
Coliform, fecal	9222D, 2006	N/A
Coliform, fecal	9223B, 2004	N/A
Coliform, fecal	Colilert-18 Quanti-Tray	N/A
e. Coli	9223B, 2004	N/A
e. Coli	m-Coliblue-24	N/A
Coliform, total	9222B, 1997	N/A
Coliform, total	9222B, 2006	N/A
Enterococci	Enterolert	N/A
Inorganic Tests		
Acidity	2310B, 2011	N/A
Alkalinity	2320B, 2011	N/A
Ammonia	4500 NH3-H, 2011	N/A
Biochemical oxygen demand	5210B, 2011	N/A
BOD, carbonaceous	5210B, 2011	N/A
Chemical oxygen demand	5220D, 2011	N/A
Chloride	4500 Cl-E, 2011	N/A
Chlorine, total residual	4500 Cl-G, 2011	N/A
Color	2120B, 2011	N/A
Cyanide, total & amenable to chlor.	4500 CN-E,G, 2011	9012B
Fluoride	4500 F-C, 2011	N/A
Hardness	2340 C, 2011	N/A
Hydrogen ion (ph)	4500 H+B, 2011	9040C
Kjeldahl and organic nitrogen	4500 N _{org} B, 2011	N/A
Nitrate	4500 NO3-F, 2011	N/A
Nitrate-nitrite	4500 NO3-F, 2011	N/A
Nitrite	4500 NO3-F, 2011	N/A
Oil and grease	1664A / 1664B	N/A
Total Organic carbon	5310B, 2011	N/A
Orthophosphate	4500 –P E, 2011	N/A
Oxygen, dissolved (probe)	4500 O-G, 2011	N/A
Phenols	420.4/9066	9065
Phosphorus, total	4500 –P E, 2011	N/A
Residue, total	2540 B, 2011	N/A
Residue, filterable (TDS)	2540-C, 2011	N/A
Residue, nonfilterable (TSS)	2540-D, 2011	N/A
Residue, settleable	2540B, 2011	N/A
Specific conductance	120.1	9050A
Sulfate	4500 SO ₄ -E, 2011	9038
Metals		
Chromium VI	3500-Cr B, 2011	7196A
Mercury	245.1 / 7470A	7471B
All other metals	200.7 / 6010B / 6010C	6010B/6010C
Organic Tests		2
Purgeable Aromatic Hydrocarbons (BTEX)	602/8021B	8021B
TPH-GRO	OK –GRO	OK – GRO
TPH-DRO	OK – DRO	OK – DRO
Volatile Organics	624.1 / 8260C	8260C
Semivolatile Organics	625.1 / 8270D	8270D



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APPENDIX VI

ACCREDITATIONS AND CERTIFICATIONS



APPENDIX VII Laboratory Equipment

Lab I.D. Balance 3 Balance 4 Balance 9 Balance 13 Balance 16 Balance 17 Balance 18 Balance 19 Centrifuge 1 Centrifuge 2 Charger 1 Chaser 1 Chiller 2 Chiller 3 Chiller 4 COD Reactor 2 **COD** Reactor 4 Concentrator 1 **Concentrator 3** Cond Meter 1 Distiller 1 Distiller 2 DO Meter 1 DO Meter 2 DO Meter 3 DO Meter 4 EasyChem 1 Flashpoint 1 Flashpoint 2 Freezer 1 Freezer 2 Freezer 3 GC2 GC3 GC4 GCMS1 GCMS1 GCMS2 GCMS2 Hot Block 1 Hot Block 2 Hot Block 3 Hot Block 5 Hot Block 6 Hot/Stir Plate 1 Hot/Stir Plate 2 Hot/Stir Plate 3

Equipment **Ohaus TS600S Precision Standard Balance Ohaus TS200S Precision Standard Balance Denver Instruments SI-402** Mettler Toledo XS403S Sartorius Top Loader S102-1S Sartorius Practum 412-1S Sartorius Secura 224-1S Sartorius Practum 224-1S Dynac II 4 place 50mL Centrifuge Damon IEC Division Clinical Centrifuge ISCO 5 Station Battery Charger Zip Scientific GC Chaser GCMS 1 **ICP** Water Recirculator Easy Dist Water Recirculator Neslab Thermoflex 900 30 Place HACH DRB 200 COD Reactor 30 Place HACH DRB 200 COD Reactor TurboVapII Concentrator TurboVapII Concentrator Orion 162 Conductivity Meter Westco Easy-Dist Westco Easy-Dist YSI 5000 DO Meter YSI 5100 DO Meter YSI Pro ODO Meter YSI Pro ODO Meter Easy Chem Plus Pensky-Martin Closed Cup Flashpoint **Cleveland Open Cup Flashpoint** Kenmore 255.29502010 (BTEX/GRO stds) Kenmore 255.29502010 (VOA stds) HotPoint HT518GBSARCC (Top of Fridge 13) HP 5890 Series II GC - FID/NPD HP 5890 Series II GC - FID/PID HP 5890 Series II GC - FID/PID HP 5973 MS HP 6890 GC HP 5890 Series II Plus GC HP 5972 MS 36 Well HotBlock SC100 Env Express 36 Well HotBlock SC100 Env Express 54 Well HotBlock SC154 Env Express **TDS Hot Block Env Express** Simple Dist Micro Hot Block Env Express White Corning Hot/Stir Plate Thermolyne Square Midget Stirrer Corning PC-351 Hot Plate/Stir Plate (Lachat Reagents) S/N C13223972 C17025644 19250963 B022039280 32950257 33350075 0033550138 33550085 None 05863 198L00563 01215590 1B1270437 112299-2842-04 01102676011210110 1195851 17110C0349 TV0207N10759 TV0613N121978 72192019 Unknown 1136 98C0635Ae 06J2459 16A103612 17L100658 Unknown 2165 10AZ-7 None None VM785832 3235A45394 3033A31773 30336A53860 US931122804 US00030542 3336A56791 3435A01882 None None None 2017TDSW192 2017MDISW138 230898057844 None None



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Hot/Stir Plate 4 Hot/Stir Plate 5 Hot/Stir Plate 6 Hot/Stir Plate 17 Hot/Stir Plate 18 IC 1 ICP 2 **ICPMS** 1 Incubator 3 Incubator 5 Incubator 7 Incubator 14 Incubator 16 Incubator 17 Lab Autosamp 1 Lab Autosamp 2 Lab Autosamp 3 Lab Autosamp 6 Lab Autosamp 7 Lab Autosamp 8 Lab Autosamp 10 Lab Autosamp 11 Lab Autosamp 12 Lab Autosamp 19 Lab Autosamp 20 Lab Autosamp 21 Lab Autosamp 22 Lachat 1 Mercury 2 Mixer 1 Mixer 2 Mixer 3 Muffle Furnace 1 Oven 1 Oven 2 Oven 3 Oven 4 Oven 5 Oven 6 pH Meter 1 pH Meter 2 pH Meter 3 pH Meter 4 Port Meter 1 Port Meter 2 Port Meter 1 Port Meter 7 Refrigerator 1 **Refrigerator 2 Refrigerator 4 Refrigerator 8 Refrigerator 9** Refrigerator 10 Refrigerator 11

Markson Silver Hot/Stir Plate 9175 Nuova Thermolvne Hot Plate Corning PC-620 Hot/Stir Plate Barnstead / Thermolyne 525C Hot/Stir Plate Fisher SP88850200 Dionex DX-100 IC Thermo Fisher ICAP 6500 Elan 9000 ICP-MS Blue M Incubator 100A Crosley Shelvador BOD Incubator Blue M Incubator 100A Fisher BOD Incubator 3720A Fisher BOD Incubator 3720A Thermo TSCOL35 Water Bath Gilson XYZ Autosampler for Lachat GCMS 1 Autosampler (HP 7683) GCMS 1 Autosampler (HP 5973) GC 2 Autosampler (HP 7673) Lachat Gilson XYZ 2100-000 Autosampler Shimadzu ASI-5000A-S-P Elan ICPMS S-10 Perkin Elmer Autosampler Perkin Elmer AS-91 Mercury Autosampler Tekmar 2050 Auto sampler EST Analytical Evolution P&T EST Analytical Centurion EST Analytical Evolution P&T EST Analytical Centurion Lachat Automated Ion Analyzer Perkin-Elmer FIMS-100 FisherBrand G-560 Single Tube Vortex Mixer Scientific Products Single Vial Mixer Lab-Line Instruments 4 Place Shaker Lindberg Muffle Furnace CMS Equatherm Gravity Oven (TDS) Blue M Stabil-Therm Fisher Isotemp 411 (TS/TSS) Gallenkamp Oven (O&G) Blue M Stabil-Therm Gravity Oven Black Precision Oven Beckman 32 pH Meter Oakton 510 pH Meter (O&G/Lachat) Orion 4 Star Fluoride Meter (Ion/pH Meter) Orion 261 (pH Meter) Fisher Accumet AP61 Portable pH Meter (Field) HACH Portable Chlorine Colorimeter Fisher Accumet AP61 Portable pH Meter (Field) Fisher Accumet AP61 Portable pH Meter (Field) Avanti 37-9RG Refrigerator (SVOA Samps) HotPoint CTX24WKB (DRO/SVOA Samps) Magic Chef MBBR440 (SVOA Stds) HotPoint CTX24GLR (DRO Prep) Frigidaire LFHT1513LW9 (Lachat/O&G Stds) Kenmore ColdSpot 106.8658281 (BTEX/GRO Samps) Sanyo SR-4310W (DRO/SVOC Stds)

1308 756990653808 230898057844 9799601030689 C302001091647919 94070171 IC65DC131719 AJ11230701H ZA-7094 06400035BF ZA-6587 300040997 300153002 300162939 279L0-B398 US04916529 US04809966 3315G08314 279D3-B703 37402517A 102S8072803 6018 Unknown EV 714121715 CENTS 433121715 EV 886092917 CENTS 530092917 A83000-1924 101S7050701 2134587 None 0696-0065 767348 1009001461 OV3-19020 129 89/04/236 OV1-2338 None 256107 64176 013571 1324933 None 030400033871 None 8107599 I/G 00748 ZV585529 IE27410026 DF569286 BA23019093 E61635509 010933431



Refrigerator 12 Refrigerator 13 Refrigerator 14 Refrigerator 15 Refrigerator 16 Refrigerator 18 Ring Bath 1 Ring Bath 2 Solid Phase 1 Solid Phase 2 Sonicator 1 Sonicator 2 Sonicator 3 Spectrometer 1 Spectrometer 2 HotPoint HTS18GBSARCC (Login) HotPoint HTS18GBSARCC (Wet Lab Stds) HotPoint HTS17CBTDRWW (VOC Samps) Kenmore 25594683010 (SVOA Extracts) Whirlpool for BOD Samples Frigidaire Gallery (O&G and Wet Lab Samples) Boekel Ring Bath, 4 Place Precision Scientific, 4 Place CPI Solid Phase Extractor CPI Solid Phase Extractor Heat Systems, Sonicator with Dual Horn L&R Ultrasonic Cleaner Fisher Scientific Sonicator Genesys 10Vis Spectrometer Thermo Fisher Spectronic Genesys-20

VM785835 VM785832 TS768069 BJX210C188910214 EWR4974354 None 01284 697091125 08010699 Unknown G1337 09419854 FM1233 2D7D142001 3SGE191005

ATTACHMENT B -GREEN COUNTY TESTING STANDARD OPERATING PROCEDURES FOR GRAB SAMPLES REVISION 7



SOP for Grab Samples Page 1 of 5 Revision 007

STANDARD OPERATING PROCEDURES

Collection of Grab Samples Standard Methods – 1060B, 2011

Revision 007 GrabSamp_007 Effective Date: 1/1/20 Supersedes Date: 11/1/14

Approved by:

Brian Duzan, Laboratory Director

24/K 12 Date

Stantion 1212

Star Yuan, Quality Assurance Officer

Date

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1.0 SCOPE AND APPLICATION:

A grab sample is a sample collected at a particular time and place which can represent only the composition of the source at that time and place. When a source is known to be fairly constant in composition over a considerable period of time and distance, then the sample may be said to be representative of that specific site at which it is collected. When a source is known to vary over time, grab samples collected at specific intervals and analyzed separately can document the extent, frequency, and duration of these variations.

2.0 SUMMARY OF METHOD:

The standard operating procedures summarizes the processes that are dealt with when collecting grab samples in relation to field sampling.

3.0 EQUIPMENT:

- 3.1 Auto-sampler.
- 3.2 Tygon tubing.
- 3.3 Strainer.
- 3.4 Battery pack or electrical source.
- 3.5 Suspension harness (if required).
- 3.6 Tool for removing manhole cover.

4.0 **REAGENTS**:

- 4.1 Alconox detergent.
- 4.2 Distilled water.
- 4.3 1:1 HNO₃.
- 4.4 Ice.

5.0 **PROCEDURE:**

Grab samples are single samples collected at a specific spot at a site over a short period of time (typically seconds or minutes). Thus, they represent a "snapshot" in both space and time of a sampling area.

5.1 **Sample Collecting Device**

5.1.1 Manual Collection of Waters

Samples may be collected manually as grabs. Samples may be collected by dipping the container into the waste stream, but only if the container does not contain a preservative that might be rinsed



out. A separate vessel may also be used to collect the sample and then transfer it to the sample bottle. If a separate vessel is used, it should be decontaminated in between sampling events. This is the preferred method when Volatile Organics are involved.

5.1.2 Mechanical Collection of Waters

Samples may be collected using the ISCO or Sigma samplers. This is preferred for waste water permits because the sampler was used to collect the composite sample and is already on-site and ready. Just remove the tubing from the composite carboy and place it into the sample bottle. Press the "pump forward" button and a single grab sample will be collected and deposited directly into the sample container. After the container is full, press the "stop" button and the sampler will stop collecting the sample. If Volatile Organics are involved, the samples should be collected manually.

5.1.3 Manual Collection of Soils or Sludges

Soil or sludges should be collected as unique grab samples unless specified to be composites. Samples should be collected with decontaminated instruments and placed in pre-cleaned sample containers.

5.1.4 **Decontamination Procedure**

- a) INITIAL RINSE To be done by the analyst immediately after use.
- b) HOT WATER & SOAP RINSE To be done with brushes or sponges, and, if necessary, clean the outside surfaces of the glassware.
- c) TAP WATER RINSE Be sure all soap is rinsed away.
- d) HNO₃ (5%) RINSE Make sure that the nitric acid touches all surfaces that could come into contact with samples or reagents.
- e) TAP WATER RINSE Be sure all the nitric acid is rinsed away.
- f) DI WATER RINSE Minimum of three times, plus at least one rinse of the outside of the glassware.
- g) ALLOW GLASSWARE TO DRY.
- h) PUT GLASSWARE AWAY Handle the glassware in such a manner as to avoid possible contamination (i.e. do not carry beakers by putting your fingers inside them).

5.2 SPECIAL INSTRUCTIONS/RESTRICTIONS/WARNINGS:

- 5.2.1 Some manholes may contain adverse or lethal atmospheres. NEVER enter a manhole or other confined space without appropriate training and direct approval from the Laboratory Director of Environmental Services.
- 5.2.2 Be sure to use proper tools to remove manhole covers in order to prevent lifting injuries.
- 5.2.3 Traffic cones, warning lights or flashers, and orange vests should be utilized to alert on-coming traffic of your presence.
- 6.0 The following section is not pertinent to this procedure. For more detailed information refer to Green Country Testing's Chemical Hygiene Plan, Quality Assurance Manual or other specific SOP's.
 - Detection limit
 - Sample preservation, Containers, Handling, and Storage
 - Interferences
 - Calibration and Standardization
 - Calculations
 - Data Assessment and Acceptance Criteria
 - Quality Control
 - Corrective Action
 - Data Validation
 - Method Performance
 - Health and Safety
 - Pollution Prevention and Waste Management

7.0 **REFERENCES:**

- 7.1 "Standard Methods for the Examination of Water and Wastewater", 22nd Edition, 1060B.
- 7.2 Standard Operating Procedure on Sampling.



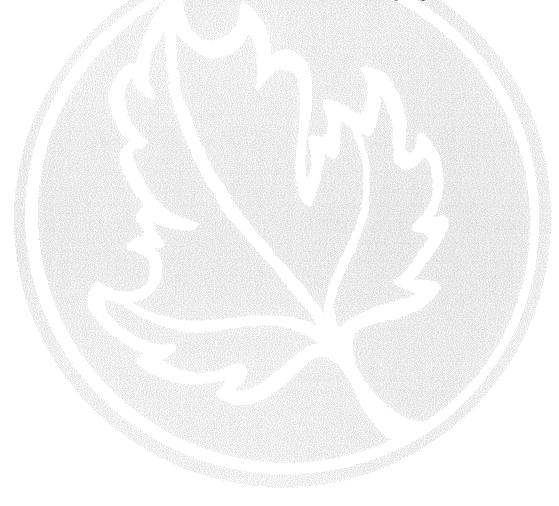


8.0 **REASON FOR SOP CHANGE:**

Yearly SOP update in section 5.1.4 regarding % Nitric in decontamination procedure.

9.0 **DEFINITIONS:**

Grab Sample- Grab samples are single samples collected at a specific spot at a site over a short period of time (typically seconds or minutes). Thus, they represent a "snapshot" in both space and time of a sampling area.



ATTACHMENT C -GREEN COUNTRY TESTING STANDARD OPERATING PROCEDURE FOR DISSOLVED OXYGEN REVISION 0



STANDARD OPERATING PROCEDURES

Dissolved Oxygen in Waters ASTMD888-09 C

Revision 000 DO_Optical_000 Effective Date: 2/7/18 Supersedes Date:

Approved by:

Brian Dazan, Laboratory Director

1124118

Star Yuan, Quality Assurance Officer

Date

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1.0 SCOPE AND APPLICATION:

The luminescence based dissolved oxygen sensor procedure is amenable to all water and wastewater matrices that are free from interferences at normal water and influent-to-treatment and final effluent wastewater concentrations.

2.0 DETECTION LIMIT:

The Practical Quantitation Limit for DO in aqueous samples is 0.1 mg/L. We typically do not conduct Method Detection Limit studies for those tests that, by nature, we are unable to conduct matrix spikes on like DO.

3.0 SUMMARY OF METHOD:

The sample is placed in a 300mL BOD bottle and analyzed while being stirred. Samples may also be analyzed in the field in-situ.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE:

Aqueous samples should be collected in an unpreserved BOD bottle and stored at 0-6°C and analyzed as soon as possible. For NPDES compliance, the sample shall be analyzed within 15 minutes of collection.

5.0 INTERFERENCES:

Sustained periods of sensor immersion in water containing high levels of chlorine dioxide may degrade sensor performance. No other interferences are known to affect the dissolved oxygen measurement.

6.0 EQUIPMENT:

YSI meter with Optical Oxygen electrode.

7.0 REAGENTS:

None

8.0 CALIBRATION AND STANDARDIZATION:

The luminescence based sensor has a built-in multipoint calibration and therefore requires no initial calibration. A single point calibration from water-saturated air is recommended when making DO measurements for regulatory reporting purposes.



9.0 **PROCEDURE:**

- 9.1 Calibration: Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of oxygen in water-saturated air. Consequently, the calibration and verification of the luminescence based sensor may be performed in air as well as water.
 - 9.1.1 Preparation of Water saturated Air Sample
 - 9.1.1.a Add ¹/₄ inch of reagent water to a clean 300mL BOD bottle and seal with stopper
 - 9.1.2.b Shake vigorously for approximately 30 seconds.
 - 9.1.3.c Allow 30 minutes for the BOD bottle and its contents to equilibrate to room temperature. The waster saturated air sample is now ready to use for calibration purposes
 - 9.1.2 Preparation of Air saturated Water
 - 9.1.2.a Add reagent water to a large glass reservoir and equilibrate to room temperature.
 - 9.1.2.b Using a steady stream of clean air, aerate the water for a minimum of 30 minutes.
 - 9.1.2.c Transfer the aerated water to clean BOD bottles until overflowing, then seal with stopper.
 - 9.1.3 Note the laboratory barometric pressure and sample temperature and use values to calculate the theoretical dissolved oxygen concentration. Analyze the prepared samples in 9.1.1 or 9.1.2 within 4 hours of preparation.
 - 9.1.4 Provide for suitable turbulent flow past the sensor cap.
 - 9.1.5 Calibration should be within 97 to 104 % of theoretical dissolved oxygen concentration.
 - 9.1.6 If the calibration verification is outside of the theoretical recovery range, re-calibrate the sensor and/or re-analyze the prepared samples.
- 9.2 Sample Measurement:
 - 9.2.1 Provide for suitable turbulent flow past the sensor cap.
 - 9.2.2 Analyze the sample.

10.0 CALCULATIONS:

The meter reads the results in mg/L.

11.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA:

The Duplicate RPD shall be within the laboratory generated control limits.



12.0 QUALITY CONTROL:

- 12.1 Blanks, laboratory control standards, and matrix spikes are not applicable to the analysis of dissolved oxygen.
- 12.2 Replicate analyses should be conducted every 10 samples or once per batch, whichever is more frequent.

13.0 CORRECTIVE ACTION:

If the duplicate RPD is outside of the acceptance limits, the data will be reported and flagged.

14.0 DATA VALIDATION:

The analyst must verify that all the QA/QC is in acceptable limits. Any data that does not meet the above criteria must be flagged if the data is to be reported. Please refer to Section 10.1 of the QA/QC Manual for more information on flagging data.

15.0 METHOD PERFORMANCE:

Precision and accuracy data are available in ASTM D888-09 C.

16.0 HEALTH AND SAFETY:

Proper eye and clothing protection must be worn at all times. Please refer to the Laboratory Chemical Hygiene plan for more detailed procedures.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT:

Please refer to the Laboratory Chemical Hygiene plan for detailed pollution prevention and waste management measures.

18.0 REFERENCES:

ASTM D888-09 C

19.0 REASON FOR SOP CHANGE:

• New SOP



20.0 DEFINITIONS:

- 20.1 Analytical Batch The set of samples prepared at the same time with appropriate quality control to a maximum of 20 samples.
- 20.2 Method Blank An aliquot of DI water treated exactly like a sample. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.
- 20.3 Laboratory Control Standard (LCS) A solution made by the laboratory by adding the analyte of interest to DI water, or purchased from a certified agency. Its purpose is to assure that the results produced by the lab remain within the acceptable limits for precision and accuracy. Equivalent to the Laboratory Fortified Blank (LFB).
- 20.4 DO Dissolved Oxygen
- 20.5 BOD Biochemical Oxygen Demand

ATTACHMENT D -GREEN COUNTRY TESTING STANDARD OPERATING PROCEDURE FOR PH IN THE FIELD REVISION 2



SOP for pH in the Field Page 1 of 6 Revision 002

STANDARD OPERATING PROCEDURES

pH in the Field Standard Methods – 4500H+B, 2011

	Revision 002 <u>pH=Field_002</u> Effective Date: 1/4/17 Supersedes Date: 1/1/16	· · ·
	Approved by:	
	12/5/1C	
	Brian Duzan, Lyboratory Director Date	
	Brian Duzan, Laborator, Director Date	
	1 an open 12-19-10	
	Star Yuan, Quality Assurance Officer Date	
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1.0 SCOPE AND APPLICATION:

- 1.1 This method is applicable to drinking, surface and saline waters, and domestic and industrial wastes. Also, multiphase and aqueous wastes where the aqueous phase constitutes at lease 20% of the total volume of the waste.
- 1.2 The corrosivity of concentrated acids and bases or of concentrated acids and bases mixed with inert substances cannot be measured. The pH measurement requires some water content.

2.0 DETECTION LIMIT:

The Practical Quantitation Limit for pH in aqueous samples is 0.1 pH units. We typically do not conduct Method Detection Limit studies for those tests that, by nature, we are unable to conduct matrix spikes on such as pH in aqueous samples.

3.0 SUMMARY OF METHOD:

The pH of the sample is determined electronically using either a glass electrode in combination with a reference potential or a combination electrode. The measuring device is calibrated using a series of standard solutions of known pH.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE:

Aqueous samples should be collected in an unpreserved container, stored at 0-6°C and analyzed as soon as possible. For NPDES compliance, the sample shall be analyzed within 15 minutes of collection.

5.0 INTERFERENCES:

- 5.1 The glass electrode in general is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or moderate (<0.1 molar solution) salinity.
- 5.2 Sodium error at pH levels >10 can be reduced or eliminated by using a low-sodium-error electrode.
 5.3 Coatings of oily matarial or
- 5.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with 10% HCl may be necessary to remove any remaining film.
- 5.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference should be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The



second source of temperature effects is the change of the pH due to changes in the sample as the temperature changes. This error is sampledependent and cannot be controlled. It should, therefore, be noted by reporting both the pH and temperature at the time of analysis. Because of these potential interferences, a temperature correcting electrode or automatic temperature compensation will be used.

6.0 EQUIPMENT:

- 6.1 pH Meter.
- 6.2 Electrode.
- 6.3 Reference electrode-a calomel, silver-silver chloride or other reference electrode of constant potential may be used.
- NOTE: Combination electrodes incorporating both measuring and reference functions are convenient and are available with solid, gel type filling materials that require minimal maintenance.
- 6.4 Temperature sensor for automatic compensation. 6.5 Beaker.
- 7.0 REAGENTS:
 - 7.1 Buffers' are purchased as solutions from commercial vendors. These commercially available solutions have been validated by comparison with NIST standards and are recommended for routine use for the calibration of the pH meter (pH 4:00, 7:00, and 10:00).
 - Standards or LCS's are purchased as solutions from commercial vendors. These commercially available solutions have been validated by comparison with NIST standards and are recommended for routine use for the calibration verification (pH 6.00 and 9.00)

8.0 CALIBRATION AND STANDARDIZATION

Certified buffers are purchased from Ricca Chemical Company or other supplier.

9.0 PROCEDURE:

The pH meter shall be calibrated and samples analyzed according to the procedures outlined in this SOP. The pH meter shall be checked with the 7 buffer at every location thru out the day.

9.1 Calibrate the pH meter according to the pH meter SOP. Each instrument/electrode system must be calibrated at a minimum of two



SOP for pH in the Field Page 4 of 6 Revision 002

points that bracket the expected pH of the samples [when possible] and are approximately three pH units or more apart. As a general rule, the pH meter shall be calibrated with the 4, 7, and 10 to cover the expected range of samples.

- 9.2 Place the sample or buffer solution in a clean, disposable, plastic beaker using sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar.
- 9.3 If field measurements are being made, the electrodes may be immersed directly in the sample stream to an adequate depth and moved in a manner to insure sufficient sample movement across the electrode-sensing element. This is indicated by drift free (<0.1 pH) readings. If the probe cannot be placed in a moving sample stream or a magnetic stirrer is not available, stir by hand at a constant rate to provide homogeneity and suspension of solids.
 9.4 After tinsing and cently blotting the solution.
 - After rinsing and gently blotting the electrodes, if necessary, immerse them into the sample beaker or sample stream and stir at a constant rate to provide homogeneity and suspension of solids. Rate of stirring should minimize the air transfer rate at the air water interface of the sample. Note and record sample pH and temperature. Repeat measurements on successive volumes of sample until values differ by less than 0.1 pH units. For strongly buffered samples and those of high ionic strength, dip the electrode in the sample for one minute. Blot dry, then immerse in a fresh portion of the sample and read the pH. With dilute or weakly buffered solutions, allow the electrode to equilibrate by immersing it in three or four successive portions of sample. Measure the pH on a fresh portion of sample.

10.0 CALCULATIONS:

9.5

pH meters read directly in pH units. Report pH to the nearest 0.01 unit and temperature to the nearest 0.01 degree C.

- 11.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA:
 - 11.1 The 6.0 and the 910 Standards (LCS's) shall be within the control limits of +/- 0.1 pH units.
 - 11.2 The 7.0 buffer analyzed at every location shall be within the control limits of +/- 0.1 pH units.
 - 11.3 The Duplicate RPD shall be within +/- 0.1 pH units or the control limits generated by the laboratory.



SOP for pH in the Field Page 5 of 6 Revision 002

12.0 QUALITY CONTROL:

- 12.1 Blanks and spikes are not applicable to the analysis of pH in aqueous samples.
- 12.2 Duplicate samples shall be analyzed daily or every ten samples, whichever is more frequent.
- 12.3 A 7.0 buffer shall be analyzed at every location.
- 12.4 Report pH values to the nearest 0.01 pH unit.

13.0 CORRECTIVE ACTION:

- 13.1 If the Standards (LCS's) are outside of the acceptance limits, they may be reanalyzed. If the second analysis is outside of the acceptance limits, the meter shall be re-calibrated and the Standards (LCS's) will be re-analyzed.
 13.2 If the duplicate RPD is outside of the acceptance limits, the second analysis of the acceptance limits.
 - 3.2 If the duplicate RPD is outside of the acceptance limits, the samples may be re-analyzed or the data will be reported and flagged.

14.0 DATA VALIDATION:

The analyst must verify that all the QA/QC is in acceptable limits. Any data that does not meet the above criteria must be flagged if the data is to be reported. Please refer to Section 10.1 of the QA/QC Manual for more information on flagging data.

15.0 METHOD PERFORMANCE:

Precision and accuracy data are available in "Standard Methods for the Examination of Water and Wastewater", 22nd Edition, 4500-H+B, 2011.

16.0 HEALTHAND SAFETY:

Proper eye and clothing protection must be worn at all times. Please refer to the Laboratory Chemical Hygiene plan for more detailed procedures. Any process that will produce a toxic vapor should be performed under a hood.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT:

Please refer to the Laboratory Chemical Hygiene plan for detailed pollution prevention and waste management measures.

18.0 REFERENCES:

"Standard Methods for the Examination of Water and Wastewater", 22nd Edition, 4500-H+ B, 2011. Electrometric Method.



19.0 REASON FOR SOP CHANGE:

Updated section 9.1 on pH calibration and 9.4 on blotting the probe.

20.0 DEFINITIONS:

- 20.1 Analytical Batch The set of samples prepared at the same time with appropriate quality control to a maximum of 20 samples.
- 20.2 Method Blank An aliquot of DI water treated exactly like a sample. The purpose is to determine if the analytes or interference's are present in the laboratory environment, the reagents, or the apparatus.
- 20.3 Buffers Buffers are purchased reagents of a known pH value and are buffered to prevent change. Buffers are used for pH calibration.
 20.3 Standards or Laboratory Control Standard (LCS) For the purchase of a H
 - 3/ Standards or Laboratory Control Standard (LCS) For the purpose of pH, the standards or LCS refer to the check standards of 6.0 and 9.0 that are analyzed after calibration to ensure accuracy.

ATTACHMENT E - GREEN COUNTRY TESTING STANDARD OPERATING PROCEDURE FOR SPECIFIC CONDUCTANCE REVISION 10



19.0 REASON FOR SOP CHANGE:

New meter.

20.0 DEFINITIONS:

- 20.1 Analytical Batch The set of samples prepared at the same time with appropriate quality control to a maximum of 20 samples.
- 20.2 Method Blank An aliquot of DI water treated exactly like a sample. The purpose is to determine if the analytes or interference's are present in the laboratory environment, the reagents, or the apparatus.
- 20.3 Laboratory Control Standard (LCS) A solution made by the laboratory by adding the analyte of interest to DI water, or purchased from a certified agency. Its purpose is to assure that the results produced by the lab remain within the acceptable limits for precision and accuracy. Equivalent to the Laboratory Fortified Blank (LFB).



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