

Quality Assurance/Quality Control Program

for
Chevron Canada Resources.

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| | |
|--|-----------|
| INTRODUCTION | 4 |
| GOAL | 5 |
| PURPOSE OF MANUAL | 5 |
| SAMPLING PLAN..... | 5 |
| SAMPLING TYPE..... | 6 |
| FIELD MONITORING..... | 6 |
| DRILLING FLUID CHARACTERIZATION | 6 |
| <i>Field Instrumental Methods.....</i> | <i>6</i> |
| SAMPLING METHODS | 11 |
| INTRODUCTION | 11 |
| OBJECTIVE..... | 12 |
| SAMPLING WASTE SUMPS..... | 12 |
| SAMPLE BOTTLES..... | 12 |
| <i>Liquid Phase</i> | <i>12</i> |
| <i>Solid Phase.....</i> | <i>13</i> |
| <i>Total Waste.....</i> | <i>13</i> |
| <i>Receiving Soil, On-Site.....</i> | <i>13</i> |
| <i>Receiving Soil, Off-site.....</i> | <i>14</i> |
| <i>Multi-Well Programs.....</i> | <i>14</i> |
| SAMPLE BOTTLE IDENTIFICATION..... | 14 |
| SAMPLING SCHEDULE | 14 |
| QUALITY CONTROL SAMPLE COLLECTION | 14 |
| CHARACTERIZATION SAMPLE COLLECTION..... | 15 |
| <i>Sampling methods</i> | <i>15</i> |
| SAMPLING REQUIREMENTS FOR SPECIFIC ANALYTICAL METHODS..... | 16 |
| <i>Total Suspended Solids.....</i> | <i>16</i> |
| REQUEST FOR ANALYSIS FORM..... | 16 |
| SAMPLE TRANSPORT AND CHAIN OF CUSTODY | 16 |
| ANALYTICAL TECHNIQUES | 17 |
| DATA MANAGEMENT | 17 |
| REPORTING AND TRACKING OF PRECISION AND ACCURACY OF MEASUREMENTS | 17 |
| DATA HANDLING | 18 |
| ANOMALOUS DATA..... | 18 |
| DETECTION LIMIT VALUES..... | 18 |
| REFERENCES..... | 19 |
| APPENDIX I..... | 20 |
| INTRODUCTION | 20 |
| SAMPLE IDENTIFICATION | 20 |
| SAMPLE IDENTIFICATION | 20 |

| | |
|---|------------|
| CHAIN-OF-CUSTODY PROCEDURES | 21 |
| OBJECTIVE:..... | 21 |
| INTRODUCTION | 21 |
| SAMPLE CUSTODY | 21 |
| DOCUMENTING OF CHAIN-OF-CUSTODY | 22 |
| <i>Sample Tag</i> | 22 |
| <i>Chain-of-Custody Record</i> | 22 |
| TRANSFER OF CUSTODY DURING TRANSPORTATION | 23 |
| FIELD RECORDS..... | 23 |
| INTRODUCTION | 23 |
| OBJECTIVE:..... | 23 |
| DOCUMENT CONTROL | 24 |
| DISPOSAL OF SAMPLES..... | 24 |
| APPENDIX II..... | 28 |
| APPENDIX III..... | 29 |
| INTRODUCTION | 29 |
| PURPOSE..... | 29 |
| PROJECT PLAN REQUIREMENTS | 29 |
| THE QUALITY CONTROL CHART: ITS CONSTRUCTION AND INTERPRETATION..... | 30 |
| Appendix I | |
| V..... | 32. |

Introduction

Environmentally sound management of spent drilling fluid, materials characterization and product stability monitoring demands a sound quality assurance program to substantiate the validity of materials sampling, process control and product performance analytical data.

A data quality assurance program is the orderly application of the practices necessary to remove or reduce errors that may occur in any spent drilling fluid remediation operation, caused by personnel, equipment, supplies, sampling procedures or analytical methodology. A quality assurance program for sampling procedures is the first step in ensuring accuracy and precision of the data.

Definition of terms:

Quality Assurance Program is the orderly application of the practices necessary to remove or reduce errors that may occur in any spent drilling fluid remediation operation, caused by personnel, equipment, supplies, sampling procedures and analytical methodology.

Quality Control Program includes all factors that could influence the results and consists of:

- use of qualified personnel/operators
- use of reliable equipment
- use of approved methodology
- use of standard operating procedures (SOP's)
- strict adherence to good laboratory practices (GLP's) and good measurement practices (GMP's)
- use of control charts
- use of appropriate calibrations and standards
- use of protocols for all critical steps
- use of protocols for special purposes
- close supervision of all operations by management.

Accuracy is the difference between the true and observed (measured) value.

Precision can be expressed as the closeness of agreement between results obtained by a particular method from the same sample, either in the same laboratory (repeatability) or in different laboratories (reproducibility).

Random errors are alterations to the observed values resulting from, for example, accidental alteration of the sample, misreading of a dial or gauge, or a one-time interference with the measuring device.

Systematic error is a consistent and relatively uniform amount of bias added to the true value by the method of observation. Examples of these kinds of errors include bent pointers on gauges, gauges that have not been zeroed properly, electrical instruments that have not been standardized and white noise addition to digital observations.

The collection of data from a spent drilling fluid remediation operation is a complex task. Error in sampling and analysis can be both systematic and random. The control of these types of error in the actual analysis resides with the analytical laboratory and is beyond the scope of this document. This document is concerned with the control of error arising from the sampling and the handling of samples at the well site or in the pilot plant. It also deals with the accuracy and the precision of measurements observed continuously through the use of on-line instrumentation in the plant, and with errors introduced through spent drilling fluid sampling.

Goal

The goal of the quality assurance/quality control program is to establish the reliability of reported data. Reliability means that the accuracy and precision of the observed data is known and consistent. This is ensured by systematically:

1. Identifying the sources of error.
2. Establishing error control processes.
3. Documenting 1. and 2.
4. Reporting performance periodically in a disciplined fashion.

Purpose of Manual

The purpose of this manual is to provide an operating policy for spent drilling fluid remediation field operations and the Chevron Canada Resources. (CHEVRON) pilot plant. The manual outlines the methods for the collection of periodic drilling waste samples, transportation of drilling waste samples to the analytical laboratory and the reporting of the accuracy and precision of data.

Sampling Plan

CHEVRON is required to establish and follow a quality assurance and quality control (QA/QC) program during its field operations and in its pilot plant. In addition to CHEVRON 's operational requirements, the Alberta Energy and Utilities Board has proposed the following monitoring program as a minimum (AUB Guide 50, Section 2):

1. Characterization:
 - a) Liquid Phase: Suspended Solids, pH, EC, Major Ions, Oil and Grease. Other parameters may be required on a site-by-site basis, for example heavy metals, Microtox[®], trace metals, radionuclides.
 - b) Solid Phase: Oil and Grease, pH, EC, Specific Gravity, Particle Size Analysis, Total Carbon, Ion Exchange Capacity, Sodium Adsorption Ratio, Soluble Ions, Ammonium, Nitrate, and Phosphate. Other parameters may be required on a site-by-site basis, for example heavy metals, Microtox[®], trace metals, radionuclides.
 - c) Gas Phase: If Volatile Organic Compounds (VOC) are present, gas phase sampling and analysis may be required.
 - d) Site factors (mass, volume, site map).
2. Pilot plant product characterization for such parameters as temperature, pH, EC, compressive and shear strength, porosity and permeability, and leachability. The same procedures described below for raw waste applies equally to samples obtained from field and laboratory leaching tests.
3. Chevron Canada Resources. has adopted a product performance monitoring program that includes the above observations and the following parameters:
 - leachate and groundwater pH, EC, turbidity, oil and grease, and chromate
 - rainfall and piezometric head
 - monolith permeability (subsurface disposal)
 - roadbed permeability (solidified waste used as road foundation material)

Additional (or no) performance monitoring requirements may be applied to particular sites, at the discretion of the regulatory agencies involved.

Sampling Type

Proper identification of samples is required to determine where they were collected in the process and what treatment processes were used in treating the drilling waste at the time of sampling.

Field Monitoring

The objectives in measuring field parameters are:

1. To ensure that the sample is representative of actual conditions and;
2. To obtain data on some of the parameters that may change between sample collection and sample analysis.

The following parameters are measured periodically:

1. piezometric head in at least four piezometers nests completed at three depths
2. leachate levels
3. temperature
4. pH
5. suspended solids in leachate
6. turbidity of leachate

These data are measured continuously and are captured and stored in a personal computer.

Drilling Fluid Characterization

This section describes the protocol to be followed for the collection, labeling, transportation and analysis of drilling fluid samples obtained at either an active well site or from a previously abandoned waste sump. Both liquid phase and solid phase sample management is discussed. In all cases, Composite sampling will be used at the well site for the collection of both liquid and solid phase samples. These samples are drawn by an automatic sampling device and composited by combining in a two liter glass container. A composite sample representing a 24-h period is considered standard in calculating the masses and volumes of spent drilling fluid and cuttings entering the waste sump.

Field Instrumental Methods

Measurement of certain parameters that may change with transportation must be measured accurately in the field. This is particularly true for sites where non-hydrocarbon liquid-phase material is present and where hydrocarbon materials contain VOC. The procedures that follow were derived from "Standard Methods for the Analysis of Water and Wastewater (16th Edition (1985), and the 18th Edition, 1992) and from the USEPA "Methods for Chemical Analysis of Water and Wastes, ES-EPA 150.1 (1983).

Liquid Phase Sampling: Temperature (SM 2510B)

Temperature is a measure of hotness or coldness on a defined scale.

Three types of thermometers are available:

- Digital (then-no-couple) thermistor
- Glass bulb mercury filled
- Bimetal strip/dial indicator

Calibration:

Whichever type of thermometer is used, it should be calibrated semi-annually against a National

Instrumentation Standards and Technology (NIST) certified thermometer.

Note: Thermistors should be checked against a mercury bulb thermometer prior to use and should agree within ± 0.5 °C.

Inspection:

All thermometers should be inspected for leaks, cracks, and/or function prior to use.

Note: A broken glass bulb-mercury filled thermometer can contaminate samples by the release of mercury vapors.

Procedures: (Make measurements in-situ when possible)

1. Clean the probe end with de-ionized water and immerse into sample.
2. Swirl the thermometer in the sample.
3. Allow the thermometer to equilibrate with the sample.
4. Suspend the thermometer away from the sides and bottom to observe the reading.
5. Record the reading in the log book. Report temperatures readings to the nearest 0.5 °C.

Note: Always clean the thermometer prior to storage and/or use.

Units:

Degrees Celsius (°C) or Degrees Fahrenheit (°F)

Conversion Formulas:

$$^{\circ}\text{F} = (9/5 \text{ } ^{\circ}\text{C}) + 32 \text{ or } ^{\circ}\text{C} = 5/9 (\text{ } ^{\circ}\text{F} - 32)$$

Liquid Phase Sampling: Hydrogen Ion Concentration (pH) (SM Method 423)

The pH is defined as the negative logarithm of the effective hydrogen-ion concentration or hydrogen-ion activity in grams equivalents per liter used in expressing both acidity and alkalinity on a scale, which ranges from 0 to 14 with 7 representing neutrality.

Meter(s) available:

Orion Model 399A

Orion SA 250 or 230A

Hydrolab Surveyor 11

YSI3530 Water Quality Monitoring System

Calibration: (Follow manufacturer's instructions with the following as a minimum)

Note: The pH of the sample to be tested should be estimated either from historical data or by using a four-color pH indicator paper or equivalent. Using this information, the two buffering points for calibration can be determined.

1. Remove the meter from storage and allow it to equilibrate to ambient temperature.
2. Use a thermometer and determine the temperature of the buffering solutions and record.
3. Select either pH 4 and pH 7 or pH 7 and pH IO solutions as described above.
4. Rinse the probe with analyte-free water and immerse it into the first buffer (pH 7) and record.

5. Rinse the probe with analyte-free water and immerse it into the second buffer and record.
6. Rinse and store the probe in a container filled with analyte-free water.

Procedures:

1. Collect a sample. Measure the temperature prior to measuring the pH.
Note: If the temperature of the sample differs by more than 2°C or approximately 4°F, refer to the manufactures instructions on how to adjust for temperature variations.
Note: When the pH meter response is slow, unstable, or non-reproducible, it may be necessary to check the conductivity. If the conductivity is lower than 20 to 30 /mhos/cm then add 1 ml of 1M potassium chloride solution per 100 ml of sample. Recheck the pH and record.
2. Immerse the probe in the sample keeping it away from the sides and bottom of the sample container. Allow ample time for the probe to equilibrate with the sample.
3. While suspending the probe away from the sides and bottom of the sample container, record the pH.
4. Rinse the probe with analyte-free water and store it in an analyte-free water **filled** container until the next sample is ready.

Operational check:

1. While in use, periodically check the pH by rinsing the probe with analyte-free water and immersing it into the pH 7 buffer solution.
2. Perform a post calibration at the end of the day and record all findings.

Units:

Units of pH are Standard Units (SU) and should be read in one-hundredths (0.01) and recorded in tenths (0. 1).

- Note:** If the pH measurements are to be used for RCRA regulatory purposes and when the pH approaches the alkaline end ($\text{pH} \geq 11.0$) of the scale, the pH measurements should be made by a qualified analyst using laboratory quality equipment to control the sample at 25°C ±0.1°C.

Liquid Phase Sampling: Electrical Conductivity (USEPA Method 150.1)

Conductivity is defined as the quality or power of conducting or transmitting.

Meter(s) available:

Wheatstone bridge meters are typically used for measuring conductivity.

Calibration:

The meter should be calibrated in accordance with the manufacturer's instructions. A two-point standard should be used to insure the accuracy of the meter. Conductivity may be affected by temperature; therefore, temperature should be read first so that appropriate adjustments can be made in accordance to the manufacturer's instructions.

1. Check and record the temperature of the standard solutions.
2. Rinse the probe with analyte-free water before immersing it in the standards solution.
3. Immerse the probe in the first standard solution and record the results.

Note: Make sure the meter is "ON".

4. Rinse the probe and immerse it into the second standard solution and record results.

Note: If the meter is not accurate to within $\pm 10\%$ of the standards, correct the problem before proceeding.

Procedures:

1. Collect the sample and check and record its temperature.
2. Correct the instruments temperature adjustment to the temperature of the sample (if required).
3. Immerse the probe in the sample keeping it away from the sides and bottom of the container. It is important that the enter portion of the probe be wetted by the sample. This will be evident when some of the sample water is seen coming out of the small weep hole.
4. Record the results in a log book.
5. Rinse probe.

Units:

Conductivity units are measured in micromhos per centimeter ($\mu\text{mhos/cm}$) at 25°C. Results should be reported to the nearest ten (10) units for readings below 1,000 $\mu\text{mho/cm}$ @ 25°C and to the nearest one hundred (100) units for reading above 1,000 $\mu\text{mhos/cm}$ @ 25°C.

Liquid Phase Sampling: Turbidity (Hach Method)

A nephelometer/turbidimeter is used in comparing the turbidity of liquids by viewing light through them and determining how much light is eliminated.

Meter(s) available:

- Hach 2100P Turbidimeter

Calibration:

1. Turn the meter "ON".
2. Rinse the sample cell 3 times with organic free or deionized water.
3. Fill the cell to the fill line with organic free or deionized water and then cap the cell.
4. Use a non-abrasive lint-free paper or cloth (preferably lens paper) to wipe off excess water and streaks.
5. Open the cover and insert the cell (arrow to the front) into the unit and close the cover.
6. Press, "READ" and wait for the 'light bulb' icon to go off. Record the reading.
7. Using the Gelex standards, repeat steps 4, 5, and 6. Record all findings (note anomalies).

Procedures:

1. Collect a specific sample or use a portion of the sample that is collected for pH, temperature, or conductivity analysis, and pour off enough to fill the cell to the fill line (approximately 3/4 full) and replace the cap on the cell.
2. Wipe off excess water and any streaks with non-abrasive lint-free paper or cloth (lens paper).
3. Place the cell in chamber of the 2100P with the arrow towards the front and close the cover.
4. Press, "READ" and wait for the 'light bulb' icon to go off. Record the reading.
5. Rinse the cell with organic-free or analyte-free water.
6. For the next sample, repeat Steps 1-5.

Operational check:

1. Periodically check the turbidity meter by using the standards provided.
2. Perform a post calibration at the end of the day and record all findings.

Units:

Turbidity measurements are reported in nephelometric turbidity units (NTUs).

Liquid Phase Sampling: Major Ions (S.M.)

Liquid Phase Sampling: Trace Metals (S.M.)

Liquid Phase Sampling: Oil and Grease (S.M.)

Liquid and Solid Phase Sampling: Microtox®

Introduction

AEUB Guide 50, Section 7, mandates the following procedures.

Toxicity bioassays of drilling wastes are used to determine if components that might be harmful to vegetation, micro-organisms, aquatic species as well as animals or humans are present, but are not detected in routine chemical analyses. The toxicity bioassay is a screen or early warning system, which will alert the analyst, field operator and Regulatory Agency that toxic constituents may be present.

Bioassays, which are suited to the testing of drilling waste material, must be responsive to small changes in concentrations of components, which are organic, metallic, organometallic or gaseous, and should not be highly dependent on major nutrient or ion concentrations. The trout bioassay has historically been used to assess the toxicity of drilling waste materials. The Microtox® test system is used to provide the industry with a more rapid, real-time measurement of acute toxicity,

The interactions and combined effects of constituents and influence of complex matrices on toxicity cannot be determined from chemical testing alone. Bioassays can be useful in predicting the influence of environmental factors on ecotoxic responses and short and long term impacts of drilling wastes. Bioassay results can also be of assistance in developing and evaluating waste treatment and disposal options. The main purposes of conducting bioassays on drilling wastes are to:

- determine the presence or absence of toxicity
- ascertain the nature, extent and probable cause(s) of toxicity
- assist in developing and evaluating waste treatment options &
- assist in making waste disposal decisions

The Microtox® and trout bioassay methods outlined here are applicable to any aqueous, water soluble (or water partitionable) material.

Sample Collection

For toxicity testing, special precautions are required in sample collection, storage and handling to prevent accidental contamination by toxic substances, and loss of toxicity due to escape of volatile components, evaporation, sorption effects, biodegradation and chemical reactions. The techniques and procedures described below must be strictly followed to obtain reliable data and results.

Sample Containers

Collect samples in new or properly washed wide mouthed borosilicate glass containers fitted with Teflon lined lids. Plastic containers should be avoided due to the presence of toxic

antioxidants, plasticizers, and mould-releasing agents in some plastics. Some plastic containers may adsorb certain toxicants and therefore cause erroneous test results.

When the use of glass containers is not feasible, use non-toxic plastic sample containers. (Samples for trout testing are frequently collected in non-toxic plastic containers because of the danger of breakage during shipping.)

If glass sample containers are reused, sampling bottles and caps must be washed with a suitable detergent and hot water. Soak bottles and caps overnight in 1 % hydrochloric acid, rinse three times with tap water, and finally rinse three times with demineralized water. Air dry bottles (inverted) and caps prior to storage. Plastic sample containers should never be reused.

No preservatives are required or added to the sampling bottles for toxicity testing.

Controls for the influence of the container on the toxic response of the test should be included in the pre-screen assay when samples are submitted in containers other than the ones prescribed above.

Sampling Procedures

For drilling waste Fluids, Clear Liquids or Total Wastes, collect a minimum of 125 ml of sample for the Microtox® test and 4 L of sample for the trout test. For solid wastes, collect 150 g of sample for the Microtox® (g test) and 4 kg of sample for the trout test.

A *Field Data Sheet* should be completed to provide information regarding the sample description, location code, sampler (person), sample date and time, sample type (grab or composite), and other pertinent remarks.

Sample Preservation and Transport

Transport samples on ice or in a refrigeration unit.

Analyze samples as soon as possible after collection to prevent unpredictable changes. If that is not possible, store samples at 4°C in a cooler with ice or a refrigerator. The recommended maximum elapsed time between sample receipt and testing is 72 hours.

Samples containing highly volatile components are to be handled as little as possible to reduce losses due to stripping.

Sample Handling Precautions

Toxicity tests by their nature, may involve the testing of hazardous materials and highly toxic materials. The handling of such samples entails potential health hazards due to exposure to chemical and biological toxicants. Appropriate safety measures, such as the wearing of protective clothing, and maintaining proper hygienic and good laboratory practices, should be utilized to minimize chance exposure and safety related problems.

Perform toxicity tests in a well ventilated area. Use appropriate and environmentally safe procedures for the disposal of tested waste samples and hazardous materials.

Sampling Methods

Introduction

To maintain and prove data integrity, it is essential that all sampling be conducted strictly according to the following protocols. What may appear to be a minor variation in method may introduce random errors into the data, which could invalidate subsequent analysis and reporting. All variances that occur must be documented on the Field Data Sheet. All

procedures and calculations below have been mandated by regulatory agencies, are based on long experience or have been adequately proven through research published in refereed journals.

Objective

- To collect a representative sample of the liquids and solids found at a client site for further processing.

Sampling waste sumps

The Alberta Energy and Utilities Board Guide 50 states: "Collection, preservation, and storage of samples are critical to the results of testing. The data is only as valid as the sample itself. In principle, you should collect samples that are free of secondary contamination and, when analyzed in the laboratory, provide results, which are representative of the source or problem under investigation. Collect a representative sample of each of the fluid and solid phases of waste from each discrete sump at a given location. Representative samples from each discrete sump are required because wastes may be the result of unique mud systems which may be disposed of by different techniques."

"The minimum distance at which samples should be obtained from the edge of the sump is one meter."

"For sumps less than or equal to 500 m², collect samples from five locations. For sumps greater than 500 m², sample one location for each 100 m² to a maximum of 10 locations. Collect a sample from the entire depth of each of the fluid and solid phases of the waste material at each location. The suction and discharge points represent two fixed sampling locations if they are identifiable."

Sample Bottles

The following steps were derived from AEUB Guide 50, 1996 Edition. Where there is any conflict with the current version of the Guide, Guide 50 instructions and interpretations are to prevail.

Liquid Phase

Sample bottles should be made of borosilicate glass since microbes tend to stick to plastic bottle walls and result in errors from bacterial growth or in the amount of suspended solids in the water. The bottle caps should also be made of Teflon for the same reasons.

If sample bottles are to be re-used, it is essential they be free of any residue from previous samples, washing materials and foreign substances. Labels must be completely removed, particularly the "magic marker" traces from the exterior of the bottles and caps. There must be no opportunity for confusing sample identity. Sample bottles should be rinsed three times with tap water, once with denatured alcohol, three with tap water and three times with distilled water in that order. Bottles should then be capped with new Teflon liners and sealed immediately.

Emulsions or Free Hydrocarbons

- Collect individual sub-samples according to the planned disposal procedure. **Include any hydrocarbon layer in the sample if the hydrocarbon will not be removed prior to disposal.**
- Prepare a composite sample with equal amounts of five sub-samples.
Portions of the composite sample should be sub-sampled into containers as follows:

- For trace element and major ion analysis, a minimum of one liter in an uncoloured plastic container.
- For toxicity assessment, one liter in an acid washed borosilicate glass container with a Teflon-lined lid.
- For oil content, one liter in a properly cleaned glass jar with a Teflon-lined lid.

Clear Water

- Collect individual sub-samples according to the planned disposal procedure.
- Prepare a composite sample with equal amounts of five sub-samples. Portions of the composite sample should be sub-sampled into containers as follows:
 - For trace element and major ion analysis, a minimum of one liter in an uncoloured plastic container.
 - For toxicity assessment, one liter in an acid washed borosilicate glass container with a Teflon-lined lid.

For total dissolved hydrocarbons, 40 ml of the composite in a septum cap glass vial. Fill the vial to overflowing and replace cap securely.

Solid Phase

- Place a minimum of two kilograms of the composite sample comprised of equal amounts of five sub-samples in an uncoloured plastic container. If a sample plastic bag is used, care must be taken to avoid loss of sample (puncture, leakage) and obscuring the sample label. Double or triple bags are recommended.
- For oil content, place one liter of the composite in a properly cleaned glass jar with a Teflon-lined lid.

Total Waste

If the Fluid and Solid phases of the sump are to be disposed of together, a Total Waste sample must be taken. A Total Waste sample is a sample that is taken from the whole depth of the Fluids and Solids at one time. **A Total Waste sample must not be made up of individually sampled Fluid and solid phases.**

- Collect individual sub-samples according to the planned disposal procedure.
- Prepare a composite sample with equal amounts of **five** sub-samples.
- Portions of the composite sample should be sub-sampled into containers as follows:
 - For trace element and major ion analysis, a minimum of one liter in an uncoloured plastic container.
 - For toxicity assessment, one liter in an acid washed borosilicate glass container with a Teflon-lined lid.
 - For oil content, place one liter in a properly cleaned glass jar with a Teflon-lined lid.

Receiving Soil, On-Site

Obtain one composite sample comprised of equal amounts of five well-distributed sub-samples from the 0-50 cm depth (or incorporation depth) in the soil horizon to be used for disposal.

Place a minimum of one kilogram of the composite subsoil in an uncoloured plastic container. Precautions must be taken to avoid loss of sample or obscuring the identification label. Double or triple bags are recommended.

Receiving Soil, Off-site

- One sampling site is required for every 8 hectares of the disposal area.
- The sampling site should be a circular area with a 10 m radius, with location coordinates known for subsequent re-sampling, if necessary.
- Ten (10) cores of topsoil (maximum 15 cm depth) are required to be taken randomly within the sample site and composite into one sample.
- The **Receiving Soil** sample site must be representative of the soil-landscape in the proposed disposal area and of the most sensitive part of the soil landscape. Convergent footslopes and toeslopes are usually the most sensitive parts of the soil-landscapes due to natural presence of high sodium and sulphate levels (in some soil-landscapes), and due to possible concentration of previous drilling sump wastes by run-off.
- For subsequent applications, the **Receiving Soil** sample site must be representative of previous drilling waste applications, as such, it must be within the area of previous coverage.
- Place a minimum of one kilogram of the composite subsoil in an uncoloured plastic container. Precautions must be taken to avoid loss of sample or obscuring the identification label. Double or triple bags are recommended.

Multi-Well Programs

Multi-well programs will be reviewed with the appropriate regulatory agency to avoid redundant sampling and analysis procedures. This information will be contained in the remediation plan for each individual site.

Sample Bottle Identification

Each sample bottle will be identified by a specific number and a letter signifying the type of sample. Sample bottle numbers must be recorded on both sample collection data sheets and the request for analysis. This will ensure that the bottle and data represent the same sample and that the sample can be tracked through the laboratory analysis.

Sampling Schedule

Samples for the periodically observed parameters will be obtained according to a schedule laid out in the remediation project plan. An example weekly schedule of sampling and sample analysis is attached in Appendix II.

Quality Control Sample Collection

Quality control samples will be limited to the collection of duplicates, fresh (unused) mud and distilled water blanks. The following definitions are used:

Replicate Sample - A time sequential sample collected at different times. Sampling during the startup phase is at a sufficiently high frequency and 24 hour composite samples are taken, it should not be necessary to collect replicate samples. Grab samples do not compare to composite samples and should not be used as replicate samples.

Duplicate Sample - A single sample collected in a suitably sized container and subsequently divided into two aliquots.

Distilled Water Blank - A sample composed of distilled water that is submitted to check sample handling, sampling procedures and sample bottle preparation.

Spiked Sample - A liquid or solid sample into which a known quantity of a known substance has been added. The reported result should be within accepted error of the method for the spiking substance for the analytical method employed.

Distilled water blanks, spiked samples and duplicate samples must be submitted under different identification numbers and collection times such that the receiving laboratory is unaware of their nature. Duplicate samples must come from the same sample batch.

A minimum of one duplicate sample, one fresh mud sample and one distilled water blank should be collected each sampling period. The sampling plan will contain the specific sites and timing of samples for each individual remediation project.

Spiked samples will be used at least once with each batch of samples submitted to each laboratory conduction analyses for the project.

A statistical control chart (Appendix III) shall be constructed for each parameter that will be periodically sampled. Control limits will be established in the project plan for all parameters to be measured.

Characterization Sample Collection

Sampling methods

The following methods are an abridged version of AEUB Guide 50 approved methods. Where there is a discrepancy between this manual and the current version of Guide 50, the Guide 50 method shall prevail.

- Prepare one composite sample of the fluid phase materials by combining equal volumes of the fluid material collected at each sample location. Composite samples should be collected in a clean container and then transferred to the appropriate sample container as soon as possible for transport. Follow the same procedure for preparing a composite solid phase sample.
- The samples should be placed in containers according to the types of analyses to be conducted. Acid washed glass jars with Teflon lids are required for samples, which will be analyzed for organic components (oil content) and Microtox[®] bioassay. Uncoloured plastic containers (bags, bottles) are required for samples, which will be analyzed for inorganic components (salts, metals, pH, others). This type of container is required to minimize the potential for sample contamination from coloured plastics.

Fluids

- Sample preparation depends on the method of analysis. As a minimum, the Fluids sample should be clarified by filtration or centrifuging to pass through a Whatman #1 (11 μ) filter paper or equivalent to remove any solids. Further clarification may be necessary.

Clear Liquids

- Samples should already be clear. However, to remove any floating particles, the sample should be clarified by filtration or centrifuging to pass through a Whatman #1 (11 μ) filter paper or equivalent. Sample preparation depends on the method of analysis chosen (see below).

Solids

- Oversaturated solids samples should be clarified by filtration or centrifuging to pass through a Whatman #1 (11 μ) filter paper or equivalent. The analysis will be done on

the clarified extract. Drying, grinding, sieving, and rewetting into a saturated paste is not required.

- Undersaturated solids samples should be brought to a saturated paste condition by adding distilled water. After standing for a minimum of four hours, the analysis will be done on the saturated paste extract.

Total Waste

- As a minimum, the sample should be clarified by filtration or centrifuging to pass through a Whatman #1 (11 μ) filter paper or equivalent. Drying, grinding, sieving, and rewetting into a saturated paste is not required.
- Oil content and metals analysis must be done on the whole sample and not on the individual phase.

Receiving Soil

- The soil sample can either be dried and a saturated paste prepared, or the sample can be handled as an undersaturated solid. Drying, grinding, sieving and rewetting into a saturated paste is not required.

Sampling Requirements for Specific Analytical Methods

The laboratory chosen for a project will supply field methods for sampling when non-standard or special analytical methods will be used. These must be appended to the project plan. One example is supplied below, for Total Suspended Solids (TSS).

Total Suspended Solids

Samples of liquids are collected by bailing or dipping from the waste sump into 2 liter glass bottles with Teflon lined caps.

A total of 2000 ml of sample is collected in a glass bottle. The sample is kept refrigerated in a cooler at 4°C and delivered to the laboratory within 48 hours for analysis.

The sample can be kept refrigerated (4°C in the dark) for no more than 7 days prior to analysis to minimize microbiological decomposition of solids.

Request for Analysis Form

The request for analysis form must accompany every sample bottle submitted for analysis. Details to be included on this form are: project, sampler's name, sample source, site sampled, date and time of sample collection, and the analyses requested. The exact form of a request for analysis will vary from laboratory to laboratory and the project plan should include a sample for the laboratory chosen for that particular project. Sample forms are attached in Appendix II, which are typical of what can be expected.

A master list of all samples collected should be prepared at the end of the monitoring period. The list should contain information on type of sample collected, purpose of sample, date and time of sample collection, sample bottle number and shipment to the receiving laboratory.

Sample Transport and Chain of Custody

All samples will be kept cool and **under no circumstances allowed to freeze**. Each sample bottle will be identified as stated. Samples will be transported in an appropriate container that will prevent breakage of the sample bottles. Those samples transported by a courier or intermediate will be shipped in a sealed container. Samples transferred to a receiving laboratory without the use of an intermediate will be delivered directly to the laboratory

supervisor or contact. Completed request for analysis forms must accompany each sample set delivered to a laboratory. Each person handling a sample should sign the request for analysis document.

Analytical Techniques

The analytical techniques, detection limits and anticipated confidence intervals used for field instrumental analysis are as follows:

Table 6-1.

| Analytical Procedures | | | |
|------------------------------|---------------|-------|------------------------|
| PARAMETER | NAQUADAT CODE | | DETECTION LIMIT |
| | Dissolved | Total | |
| pH | 10301L | | 0.01 pH units |
| Temperature | | | 0.01 degrees C |
| Electrical Conductivity | | | Varies with meter type |
| Turbidity | 02074L | | 0.1 NTU |

All procedures should conform to a standard NAQUADAT code and an effort will be made to limit all analyses to the techniques listed in Table 6-1. This precaution should minimize the possibility that variations might arise as a result of differences in analytical techniques. Particular clients may have different requirements. These must be documented in the project plan.

Data Management

All data is stored in the computer with periodic data backups. Data are prepared in graphic form for the reports to the client and/or regulatory agency.

Hard copies of data from external laboratories are received and are permanently kept in files at CHEVRON Headquarters. The data are consolidated into reports, which are forwarded to the client and/or regulatory agencies. The exact form of the report must be specified in the project plan and will vary from client to client.

The QA/QC data will be reviewed immediately upon receipt of the analytical results. The review should include an evaluation of the distilled water blanks to determine the presence of consistently spurious data and an evaluation of these data to assess whether or not they are valid. Results from duplicated samples should also be compared and plotted on a control chart (Appendix III). Outliers and anomalous values should be flagged and annotated on the control chart.

Reporting and Tracking of Precision and Accuracy of Measurements

The methodology used to analyze the water quality data will employ small sample statistics and will include the following:

1. Estimation of descriptive statistics (mean, count, maximum, minimum, standard deviation of the mean and confidence interval of the mean).
2. Standard control chart to determine tolerance limits for each determination.
3. Evaluation of the presence of significant statistical differences between individual data sets by both parametric and non-parametric methods. A confidence interval approach may also be adopted.
4. Miscellaneous analyses as required.

Data Handling

The results of the parametric tests can be summarized in a table. Those parameters where a difference in the two data sets was noted should be graphically plotted to further evaluate the data. The data can then be plotted as a concentration/value against the sample number. This procedure allows for quick visual evaluation of the data and an estimation of the cause of the differences in the data sets. Those parameters for which the differences in the data sets were due to either patterns within the individual data or anomalous data can be easily identified. The differences in the data sets might be attributable to any of several causes:

1. linear trends
2. general trends
3. anomalous data or outliers
4. potential impacts of system operation

Anomalous Data

There are several reasons why an individual data point or set of data points may be considerably different than the historical data for the particular parameter in question. These include:

1. a contaminant;
2. inconsistent sampling, sample handling, or analytical technique;
3. true, but extreme concentrations.

The methodology to identify an individual data point as anomalous or an outlier is that described by the USEPA (1989):

$$T_n = \frac{(X_n - \bar{X})}{S}$$

Where:

T_n is a test statistic

X_n is the data point in question

If the absolute value of T_n exceeds a critical value (USEPA, 1989; Table 3, Appendix E) then there is sufficient evidence to indicate that the data point is a statistical outlier.

The decision to exclude an outlier from the data is left to the discretion of the individual analyst. There must, however, also be sufficient information available to demonstrate that the cause of the anomalous value is not a contaminant event before such an exclusion is warranted.

Detection Limit Values

The concentrations of individual parameters may, at certain times, be less than the detection limit for the individual analyses. While there are a variety of methods with which below detection limit data can be dealt, there is no general procedure which is appropriate to all situations.

Concentrations that are less than the detectable limit for the analyses performed will be handled, statistically, as though they were equivalent to the detection limit reported.

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Appendix I

Chain of Custody:

SAMPLE CONTROL, FIELD RECORDS, AND DOCUMENT CONTROL

Introduction

All sample identification, chain-of-custody records, receipt for sample forms, and field records should be recorded with waterproof, non-erasable ink. If errors are made in any of these documents, corrections should be made by crossing a single line through the error and entering the correct information. All corrections should be initialed and dated. If possible, all corrections should be made by the individual making the error.

If information is entered onto sample tags, logbooks, or sample containers using stick-on labels, the labels should not be capable of being removed without leaving obvious indications of the attempt. Labels should never be placed over previously recorded information. Corrections to information recorded on stick-on labels should be made as stated above.

Following are definitions of terms used in this section:

Project Leader: The individual with overall responsibility for conducting a specific field investigation in accordance with this SOP.

Field Sample Custodian: Individual responsible for maintaining custody of the samples and completing the sample tags, Chain-of-Custody Record, and Receipt for Sample form.

Sample Team Leader: An individual designated by the project leader to be present during and responsible for all activities related to the collection of samples by a specific sampling team.

Sampler: The individual responsible for the actual collection of a sample.

Transferee: Any individual who receives custody of samples subsequent to release by the field sample custodian.

Laboratory Sample Custodian: Individual or their designee(s) responsible for accepting custody of samples from the field sample custodian or a transferee.

One individual may fulfill more than one of the roles described above while in the field.

Sample Identification

PERFORMANCE OBJECTIVES:

- To accurately identify samples and evidence collected.
- To adequately insure that chain-of-custody was maintained.

Sample Identification

The method of sample identification used depends on the type of sample collected. Samples collected for specific field analyses or measurement data are recorded directly in bound field logbooks or recorded directly on the Chain-of-Custody Record, with identifying information, while in the custody of the samplers. Examples include pH, temperature, and conductivity. Samples collected for laboratory analyses are identified by using standard sample tags or labels (Figure I-1) which are attached to the sample containers. The sample tags are sequentially numbered and are accountable documents after they are completed and attached to a sample. The following information shall be included on the sample tag using waterproof, non-erasable ink:

- Project number;

- Field identification or sample station number;
- A date and time of sample collection;
- Designation of the sample as a grab or composite;
- Type of sample (water, drilling waste, leachate, soil, sediment, etc.) and a very brief description of the sampling location;
- The signature of either the sampler(s) or the designated sampling team leader and the field sample custodian (if appropriate);
- Whether the sample is preserved or unpreserved;
- The general types of analyses to be performed (checked on front of tag); and
- Any relevant comments (such as readily detectable or identifiable odor, color, or known toxic properties).

Occasionally, it is necessary to obtain recorder and/or instrument charts from analytical equipment, flow recorders, etc., during field work. Mark the charts and write the following information on these charts while they are still in the instrument or recorder

- Starting and ending time(s) and date(s) for the chart.
- Take an instantaneous measurement of the media being measured by the recorder. The instantaneous measurement shall be entered at the appropriate location on the chart along with the date and time of the measurement.
- A description of the location being monitored and any other information required to interpret the data such as type of flow device, chart units, factors, etc.

All of the above information should be initialed by the field supervisor or sampler. After the chart has been removed, the field supervisor or sampler shall indicate on the chart who the chart (or copy of the chart) was received from and enter the date and time, as well as the supervisor's initials.

Chain-of-Custody Procedures

OBJECTIVE:

- To maintain and document the possession of samples from the time of collection until the data derived from the samples is received by the client.

Introduction

Chain-of-custody procedures are comprised of the following elements; 1) maintaining sample custody and 2) documentation of samples for future recovery. To document chain-of-custody, an accurate record must be maintained to trace the possession of each sample from the moment of collection to its delivery to the client.

Sample Custody

A sample is in custody when-

- It is in the actual possession of an employee or contractor of CHEVRON ;
- It is within view of an employee or contractor of CHEVRON , after having been in that person's physical possession;
- It was in the physical possession of an investigator and then they secured it to prevent tampering; and/or

- It is placed in a secure place (i.e. a locked closet, safe or other restricted access location).

Documenting of Chain-of-Custody

Sample Tag

A sample tag or label (Figure I - 1) must be completed for each sample using waterproof, non-erasable inks specified above.

Chain-of-Custody Record

The field Chain-Of-Custody Record (Figure I-2) is used to record the custody of all collected and maintained by CHEVRON employees or contractors. All sample sets shall be accompanied by a Chain-Of-Custody Record. This Chain-Of-Custody Record documents transfer of custody of samples from each person in the chain, from the sampler to the laboratory, or other organizational elements. To simplify the Chain-of-Custody Record and eliminate potential litigation problems, as few people as possible should have custody of the samples or physical evidence during the investigation. The Chain-Of-Custody Record also serves as a sample logging mechanism for the laboratory sample custodian. A Chain-of-Custody Record will be completed for all samples collected. A separate Chain-of-Custody Record must be used for each final destination or laboratory used.

The following information must be supplied to complete the field Chain-Of-Custody Record.

- The project identifier.
- The project name.
- All samplers and sampling team leaders (if applicable) must sign in the designated signature block.
- The sampling location identifier, date, and time of sample collection, and sample type, must be included on each line. One sample should be entered on each line and a sample should not be split among multiple lines.
- If multiple sampling teams are collecting samples, the sampling team leader's name should be indicated in the "Tag No./Remarks" column.
- If the individual serving as the field sample custodian is different from the individual serving as the project leader, the field sample custodian's name and company affiliation should be recorded in the "Remarks" section in the Chain-of-Custody Record. The Remarks section may also be used to record waybill numbers, registered or certified mail serial numbers, or other pertinent transportation information.
- The total number of samples must be listed in the "Total Containers" column for each site. The number of individual sample containers for each analysis must also be listed. There should not be more than one sample type per sample. Required analyses should be circled or entered in the appropriate location as indicated on the Chain-of-Custody Record.
- The tag/label numbers for each sample and any needed remarks are to be supplied in the "Tag No./Remarks" column.
- The sample custodian and subsequent transferee(s) should document the transfer of the samples listed on the Chain-of-Custody Record. The person who originally relinquishes custody should be the sample custodian. Both the person relinquishing the samples and the person receiving them must sign the form. The date and time that this occurred should be documented in the proper space on the Chain-of-Custody Record.

- Usually, the last person receiving the samples or evidence should be the laboratory sample custodian or their designee(s).

The Chain-of-Custody Record contains an unbroken sequence of serial numbers. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain of-custody should be evaluated based upon its inclusion of all of the above information in a legible format.

Samples should not be accepted from other sources unless the sample collection procedures used are known to be acceptable, can be documented, and the sample chain-of-custody can be established. If such samples are accepted, a standard sample tag containing all relevant information and the Chain-Of-Custody Record shall be completed for each set of samples.

Transfer of Custody During Transportation

- Samples shall be properly packaged for shipment in accordance with the procedures specified in the project plan. These will change according to the client's needs and regulatory agency requirements.
- All samples shall be accompanied by the Chain-Of-Custody Record. The original and one copy of the Record will be placed in a plastic bag inside the secured shipping container if samples are shipped. When shipping samples via common carrier, the "Relinquished By" box should be filled in; however, the "Received By" box should be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-Custody Record. One copy of the Record must be retained by the project leader. The original Chain-of-Custody Record must be transmitted to the project leader after the samples are accepted by the laboratory. This copy will become a part of the project file.
- If sent by common carrier, a Bill of Lading or manifest should be used. Receipts from shall be retained as part of the documentation of the chain-of-custody. The manifest, bill of lading or other transfer document from a common carrier or courier shall be recorded in the remarks section of the Chain-Of-Custody Record.

Field Records

Introduction

Careful documentation of field and laboratory activities is a measure of both the reliability of the Company in the eyes of clients and regulatory agencies. Properly maintained and easily accessible records make follow-up work and auditing quick and painless. This section describes proven techniques for record keeping.

OBJECTIVE:

- To accurately and completely document all field activities.

Each project will have a dedicated logbook. The project leader's name, the sample team leader's name (if appropriate), the project name and location, and the project identifier must be entered on the inside of the front cover of the logbook. It is recommended that each page in the logbook be numbered and dated. The entries should be legible and contain accurate and inclusive documentation of an individual's project activities.

Since field records are the basis for later written reports, language should be objective, factual, and free of personal feelings or other terminology, which might prove inappropriate. Once completed, these field logbooks become accountable documents and must be maintained as part of the project files. All aspects of sample collection and handling, as well

as visual observations, shall be documented in the field logbooks. The following is a list of information that should be included in the logbook:

- sample collection equipment (where appropriate);
- field analytical equipment, and equipment utilized to make physical measurements shall be identified;
- calculations, results, and calibration data for field sampling, field analytical, and field physical measurement equipment;
- property numbers of any sampling equipment used, if available;
- sampling station identification;
- time of sample collection;
- description of the sample location;
- description of the sample;
- who collected the sample;
- how the sample was collected;
- diagrams of processes;
- maps/sketches of sampling locations; and
- weather conditions that may affect the sample (e.g., rain, extreme heat or cold, wind, etc.)

Document Control

The term document control refers to the maintenance of inspection and investigation project files. All project files shall be maintained in accordance with CHEVRON company guidelines. All documents as outlined below shall be kept in project files. Project staff may keep copies of reports in their personal files, however, all official and original documents relating to remediation projects shall be placed in the official project files. The following documents shall be placed in the project file, if applicable:

- copy of the CHEVRON /Client contract;
- copy of the study plan;
- original Chain-Of-Custody Records and bound field logbooks;
- records obtained during the project;
- complete copy of the analytical data and letters transmitting analytical data to the client and to regulatory agencies;
- official correspondence received by or issued by the company relating to the project;
- photographs and negatives associated with the project;
- one copy of the final report and transmittal letter(s); and
- relevant documents related to the original remediation activities or follow-up activities related to the project.

Under no circumstances are any inappropriate personal observations or irrelevant information to be filed in the official project files. The project leader shall review the file at the conclusion of the project to ensure completeness.

Disposal of Samples

Disposal of samples is conducted on a case-by-case basis. Before samples which have been analyzed are disposed, laboratory personnel shall contact the project leader or his/her supervisor in writing, requesting permission to dispose of the samples. The samples will not

be disposed of until the client and the project leader or his/her supervisor completes the appropriate portions of the authorization letter, signs, and returns the letter to the laboratory, specifically giving them permission to dispose of the samples.

- Internal quality assurance samples are routinely disposed after the analytical results are reported. The laboratory does not advise the CHEVRON Quality Assurance Supervisor of the disposal of these samples.

| Sample Receipt | | | | | | |
|--------------------------|--------------|----------------|----------------|----------------------------|------------------|----------------|
| Project ID: | | Client: | | Drillsite Name: | | |
| Site Supervisor: | | | | Location (LSD/Twp/Rge/Mer) | | |
| Sample Location | Date Sampled | Time Sampled | Type of Sample | # of Containers | Tag/Label Number | Remarks |
| | | | | | | |
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| | | | | | | |
| | | | | | | |
| Courier/Freight Company: | | Date Received: | | Laboratory Supervisor: | | Date Received |
| (Signature) | | Time Received: | | (Signature) | | Time Received: |

Figure I-2

Appendix II
Sample Collection Plans and Report Examples

Appendix III

Example Control Chart for Analytical Results

Introduction

Control charts are used to detect systematic errors entering a data set over time that introduce false trends. The usual source for these errors over time is the drift of equipment baselines. Another source may be a change in sampling routine, which would tend to cause a step increase or decrease in derived values.

Random excursions over time may result from bad sampling technique, noise in instrumentation derived from an external source (i.e. nearby lightning strikes or power lines), or some other change in the measurement environment that is unrelated to the phenomenon being observed.

Purpose

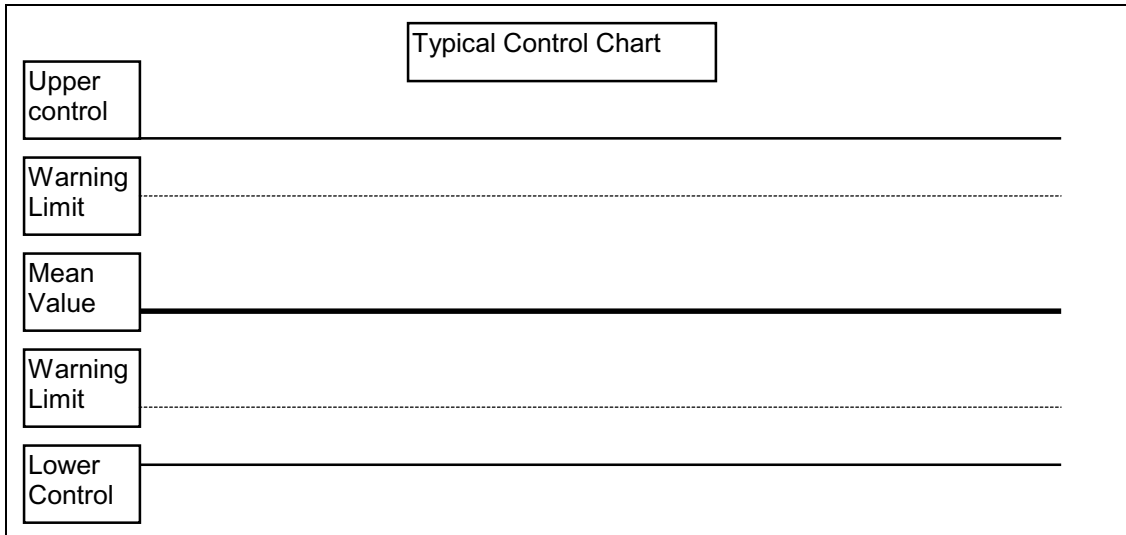
1. Criterion for confirming Statistical Control or its lack.
2. Method for identifying assignable causes.
3. Basis for confidence limits for data

Project Plan Requirements

The project plan must always establish the site and quality specifics of quality control. The following issues must be addressed:

1. *Order of sampling*
 - **Sequence:** Are there a series of sites (for example a battery of wells or a series of batteries within a field) or a single site? Are there logistical reasons for sampling them in a particular order, or can they be sampled randomly? At each site, is the rig still on site and making hole? Has the site been abandoned for long? How large is the sump? Is a pattern of observation points necessary?
 - **Time:** Are there reactions in progress that demand simultaneous sampling at several points in a sump? How often should each sampling point be observed? Are winter samples different from summer samples? Do samples separate by phase during transportation?
2. *Grouping of samples*
 - **Place:** Is there a consistent variation in parameter values across a field or well-to-well? Does mud formulation vary significantly from formation to formation down the hole? Does a given sump vary in composition vertically, or is it well homogenized?
 - **Source:** Are there several operators involved? Are all holes completed in the same formation? Were drilling problems encountered.
 - **Test Conditions:** What are the consequences to data quality and integrity of the following issues:
 - ⇒ Measurement Variables
 - ⇒ Equipment
 - ⇒ Operators
 - ⇒ Weather conditions
 - ⇒ Site conditions

The Quality Control Chart: Its Construction and Interpretation



The performance of each measurement system must be demonstrated through the repeated measurement of homogeneous and stable control samples in a planned, repetitive process. The data so generated is plotted as a control chart (example above) to indicate whether or not the measurement system is in a state of statistical control.

The control chart contains five fixed elements:

- The mean value of the parameter being observed
- An upper warning limit
- A lower warning limit
- A an upper control limit
- A lower control limit.

The vertical axis of the chart represents the magnitude of difference between a known standard and the value obtained through a particular analytical procedure (for example, a 7.00 pH standard and the value read from a particular pH meter whose electrode is immersed in a pH 7.00 buffer). The horizontal axis may be time (if measurements are periodic) or observation number (if a number of readings are taken simultaneously by different observers and instruments). Remember: a quality control chart tracks repeated measurements of a standard or a control sample (a spiked sample or a blank) over time or within a group of samplers, analytical laboratories or instruments. There will always be some random interference in a measurement system that cannot be corrected. Therefore, a steady track of zero differences is highly unlikely. The goal is to minimize variances and eliminate systematic variances.

If the instrument or method is unbiased, all results will closely follow the mean value line. If bias exists, the readings will fall above the mean value line if the instrument or method overestimates the parameter, or below the line if it underestimates the parameter.

Upper and lower warning limits are usually one standard deviation above and below the mean. Most methods are reliable within one standard deviation, but if they exceed that limit the data is to be considered unreliable and should be rejected. When a consistent drift toward a warning limit is a signal to project management that there is a problem with the observation method and corrective action is required.