

Laboratory Procedures for Analysis of Exploration & Production Waste

**Louisiana Department of Natural Resources
Office of Conservation
Injection and Mining Division**

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The Laboratory Procedures in this manual are to be utilized in testing E&P wastes for the parameters required in LAC 43:XIX.Subpart 1.

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INTRODUCTION

Following implementation of oilfield pit regulations on January 20, 1986, the Louisiana Department of Natural Resources, Office of Conservation received many questions concerning clarification of oilfield waste testing procedures for data required by LAC 43:XIX.Subpart 1.Chapter 3 and Chapter 5 (formerly Statewide Order No. 29-B, Section XV, Paragraphs 2 and 13). As a result of these questions, it became obvious that lab procedures needed to be standardized in order to improve the quality of the test results and to create reproducible data.

To meet that objective, the Office of Conservation (OC) assembled an ad-hoc committee of qualified laboratory personnel to discuss specific problems and to assist OC in adopting guidelines and/or policies for the exploration and production (E&P) waste testing procedures required by regulation.

The first ad-hoc committee was assembled and met on March 18, 1987. During subsequent meetings, lab procedures were clarified in an attempt to reduce or eliminate errors in results. At the conclusion of these meetings, the lab manual was finalized and first printed in August 1988. The manual was subsequently revised in January 1989 and reprinted in July 1995.

An Emergency Rule implemented on May 1, 1998 required characterization and analytical testing of E&P Waste received by commercial facilities regulated by OC. As a result, a second ad-hoc committee was formed to advise OC on updating the lab manual in preparation of any subsequent revisions to the rule. OC published proposed revisions to LAC 43:XIX.Subpart 1.Chapter 5 on August 20, 2001 and conducted a public hearing on September 27, 2001. Subsequently, members of the second ad-hoc committee reconvened to finalize revisions to the lab manual in accordance with final changes to LAC 43:XIX.Subpart 1.Chapter 5 which were promulgated on November 20, 2001.

Minor revisions were made to the lab manual in May 2005 and November 2011. The May 2005 revision was issued to clarify applicability of Department of Environmental Quality laboratory accreditation for analytical data generated for compliance with the requirements of LAC 43:XIX.Subpart 1. The November 2011 revision updated methods listed in the Analytical Methodology Reference Tables for pH, TSS, TDS and Reactive Sulfides and provided further guidance for use of the Sample Preparation method for SAR, ESP, CEC, pH and LAC 43:XIX.Subpart 1.Chapter 3 heavy metals digestion protocols.

This manual is a compilation of procedures for oilfield waste sampling and analyses which resulted from the work of these committees. The Office of Conservation wishes to express its appreciation to those who took part in these endeavors by contributing their time, by providing laboratory services and by sharing their expertise.

ANALYTICAL METHODOLOGY REFERENCE TABLE

LAC 43:XIX.Subpart 1

Parameter	Method
<u>Soils / Sediment / Sludges / Reusable Material</u>	
pH	SW-846 9045C, EPA 150.2^
TPH (Total Petroleum Hydrocarbons)	SW-846 1664 HEM/SGT / SW-846 9071
Leachable TPH Test	LDNR Lab Procedures for Analysis of E&P Waste
Leachable Chlorides Test	LDNR Lab Procedures for Analysis of E&P Waste
Electrical Conductivity (EC)	LDNR Lab Procedures for Analysis of E&P Waste
Sodium Absorption Ratio (SAR)	LDNR Lab Procedures for Analysis of E&P Waste
Exchangeable Sodium Percentage (ESP)	LDNR Lab Procedures for Analysis of E&P Waste
Cation Exchange Capacity (CEC)	LDNR Lab Procedures for Analysis of E&P Waste
Benzene	SW-846 8021
TCLP Benzene	SW-846 8021/1311
NORM	EMSL-LV-0539-17@
Reactive Sulfide	SW-846 9030B/9034
<u>Total Metals and/or Leachable Metals</u>	
TCLP Extraction	SW-846 1311
Arsenic (As)	SW-846 6010/6020/7000
True Total Barium (Ba)	LDNR Lab Procedures for Analysis of E&P Waste
Barium (Ba)	SW-846 6010/6020/7000
Cadmium (Cd)	SW-846 6010/6020/7000
Chromium (Cr)	SW-846 6010/6020/7000
Copper (Cu)	SW-846 6010/6020/7000
Lead (Pb)	SW-846 6010/6020/7000
Mercury (Hg)	SW-846 7470/7471
Molybdenum (Mo)	SW-846 6010/6020/7000
Nickel (Ni)	SW-846 6010/6020/7000
Selenium (Se)	SW-846 6010/6020/7000
Silver (Ag)	SW-846 6010/6020/7000
Sodium (Na)	SW-846 6010/6020/7000
Zinc (Zn)	SW-846 6010/6020/7000
Soluble Cations (Na, Ca, Mg)	LDNR Lab Procedures for Analysis of E&P Waste
Soluble Anions (Cl, CO ₃ , HCO ₃ , SO ₄)	SW-846 9056/9253, SM 2320B*

ANALYTICAL METHODOLOGY REFERENCE TABLE (cont'd)
LAC 43:XIX.Subpart 1

Parameter	Method
<u>Groundwater / Surface Water</u>	
pH	SW-846 9040B, EPA 150.2^
Electrical Conductivity (EC)	SM 120.1*
Total Petroleum Hydrocarbons (TPH)	SW-846 1664 HEM / SGT
Total Benzene	SW-846 8021
Total Suspended Solids (TSS)	SM 2540-D*
Total Dissolved Solids (TDS)	SM 2540-C*
Chlorides (Cl)	SW-846 9056/9253
Sodium Adsorption Ratio	SW-846 6010/6020/7000
Reactive Sulfide	SW-846 9030B/9034
NORM	EMSL-LV-0539-17@
<u>Total Metals</u>	
Arsenic (As)	SW-846 6010/6020/7000
Barium (Ba)	SW-846 6010/6020/7000
Cadmium (Cd)	SW-846 6010/6020/7000
Chromium (Cr)	SW-846 6010/6020/7000
Copper (Cu)	SW-846 6010/6020/7000
Mercury (Hg)	SW-846 7470/7471
Molybdenum (Mo)	SW-846 6010/6020/7000
Nickel (Ni)	SW-846 6010/6020/7000
Lead (Pb)	SW-846 6010/6020/7000
Silver (Ag)	SW-846 6010/6020/7000
Sodium (Na)	SW-846 6010/6020/7000
Selenium (Se)	SW-846 6010/6020/7000
Zinc (Zn)	SW-846 6010/6020/7000

SW-846 methods taken from EPA "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"

@ Method taken from EPA "Radiochemical Analytical Procedures for Analysis of Environmental Samples"

* Method taken from AWWA, APHA, WEF "Standard Methods for the Examination of Water and Wastewater"

^ Methods taken from EPA "Methods for Chemical Analysis of Water and Wastes"

NOTES:

- 1) Approved methods are from the latest revision of the referenced documents.
- 2) Methods listed may change due to agency updates, etc.
- 3) All testing must be performed by DEQ LELAP accredited laboratories.
- 4) Confirmation of detectable quantities of Benzene by EPA Method 8260 is suggested.
- 5) Sample collection procedures for all groundwater and surface water analysis must follow EPA approved sampling guidelines (e.g., USEPA RCRA Sampling Procedures Handbook, USEPA RCRA Groundwater Monitoring Technical Enforcement Guidance Document, etc.).
- 6) Procedures used for the acquisition of samples for the analysis of any volatile organic compounds in soil and sludge matrices should minimize the loss of volatile organic compounds during field sampling events. EPA or LDEQ approved field sampling methodology is recommended. Land treatment cells may be sampled in accordance with the sampling guidelines set forth herein.

EXPLORATION & PRODUCTION WASTE
LABORATORY PROCEDURES

SAMPLE PREPARATION

1.0 SCOPE AND APPLICATION

1.1 This method is used to prepare samples for analysis by the protocols listed below.

1.1.1 Sodium Adsorption Ratio

1.1.2 Exchangeable Sodium Percentage

1.1.3 Cation Exchange Capacity

1.1.4 pH

1.1.5 Heavy metals digestion (LAC 43:XIX.Subpart 1.Chapter 3 only)

2.0 SUMMARY OF METHOD

2.1 The sample is homogenized, dried at 38 C* and ground prior to the individual analyses. *Drying temperatures up to 105 C may be necessary to prepare samples of oily E&P Waste material for the analytical protocols listed in 1.1. Any increase in the drying temperature above 38 C must be clearly explained on the analytical report of results for each of the protocol listed in 1.1. Increases in oven drying temperatures above 38 C should only be used when necessary to condition a sample to a state suitable for performing the analysis listed in 1.1 to achieve the most reliable and accurate analytical results.

3.0 INTERFERENCES

3.1 Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Oven capable of maintaining a temperature of 105 C (+/- 2).

4.2 Grinding apparatus

4.3 Drying pans

4.4 Balance

5.0 PROCEDURE

5.1 Homogenize the sample thoroughly.

5.2 Remove debris such as rocks and plant matter.

5.3 Weigh a pan to the nearest tenth of a gram that is large enough to hold between 100 and 200 grams of sample.

5.4 Add approximately 100 grams of sample to the pan and weigh the pan plus its contents to the nearest tenth of a gram.

5.5 Place the pan and its contents in an oven at 38 C* until a constant weight is achieved. Moisture content will be determined on this sample, however, additional material may need to be dried so that enough material is available for analysis.

5.6 Cool the sample to room temperature and weigh the pan plus its contents. From this information calculate the moisture content.

5.7 Grind the material so that it will pass a 2mm sieve. The sample is now ready for the appropriate analyses.

6.0 **PROCEDURE FOR HYDROPHOBIC MATERIAL**

6.1 This procedure is used if after performing steps 5.0 through 5.6 the sample exhibits any of the following physical characteristics.

6.1.1 Visible blobs of oil and grease.

6.1.2 The sample presses into a single damp looking mass when the sample is crushed with a mortar and pestle.

6.1.3 The sample leaves an oily mark when pressed between two pieces of filter paper.

6.1.4 The sample feels damp when pinched between the fingers.

6.2 Place the sample in a muffle furnace at 250 C for 1 hour.

6.3 Increase the temperature of the muffle furnace to 350 C and ash the sample for an additional 7 hours.

6.4 Cool the sample and grind it to pass a 2mm sieve. The sample is now ready for analysis.

7.0 **REFERENCES**

7.1 Rhoades, J.D. 1996. D.L. Sparks (ed.) Methods of Soils Analysis. Part 3. Chemical Methods. SSSA Book Series No. 5. ASA Madison, WI.

SATURATED PASTE

1.0 SCOPE AND APPLICATIONS

1.1 The saturation point can be applied to soils and most solid wastes. It is used to determine the moisture concentration at which electrical conductivity must be reported. Electrical conductivity is determined on a 1:1 water extract and the reading is corrected to a conductivity at the saturation point. See the conductivity section for the calculation.

2.0 SUMMARY OF METHOD

2.1 Water is added to a known amount of sample until the point where no more water can be absorbed and additional water would result in free water on the surface.

3.0 INTERFERENCES

3.1 Stirring the soil water mixture excessively while attempting to reach the saturation point results in over saturation of the sample. Generally, a saturation point greater than 80% indicates the sample has been over saturated.

4.0 SAMPLE PREPARATION

4.1 See the sample preparation section of this manual.

5.0 APPARATUS

5.1 Container of 250 ml. capacity or greater, such as a cup or beaker.

5.2 Buchner funnels, filter rack or flask, filter paper, vacuum pump, extract containers such as test tubes or 1 oz. bottles.

6.0 PROCEDURE FOR PASTE PREPARATION

6.1 Weigh to the nearest tenth of a gram at least 50 grams of sample. Transfer the sample to a beaker and prepare the saturated paste by adding distilled water to the sample with a minimum of stirring to prevent puddling of the sample (over saturation). The sample-water mixture is consolidated from time to time during the stirring process by tapping the container on the workbench.

6.2 At saturation, the paste glistens as it reflects light, flows slightly when the container is tipped, and the paste slides freely and cleanly off the spatula for all samples but those with a high clay content.

6.3 After mixing, the sample should be covered and allowed to stand for approximately

an hour, and then the criteria for saturation should be rechecked. Free water should not collect on the sample surface nor should the paste stiffen markedly or lose its glistening appearance on standing.

6.4 If the paste does stiffen or lose its glisten, remix with more water.

6.5 Because samples puddle most readily when worked at moisture contents near field capacity, sufficient water should be added immediately to bring the sample nearly to saturation. If the paste is too wet, additional dry sample may be added. Special precautions must be taken with peat and muck soils and with samples of very fine and very coarse texture.

6.6 Let the paste stand for 12 to 24 hours before extracting to allow sparingly soluble salts to reach an equilibrium.

6.7 Take a subsample of the paste and dry the sample in an oven at 105 C and use the weight loss to determine the saturation percentage (amount of water added) as follows:

$$s = (w-d)/d$$

Where:

w = wet weight of sample

d = dry weight of sample

7.0 PASTE EXTRACT COLLECTION

7.1 Transfer the saturated sample paste to the filter funnel with a pre-wet filter paper in place and apply vacuum. Collect the extract in a bottle or test tube.

7.2 If the initial filtrate is turbid, it can be refiltered through the sample or discarded.

7.3 Vacuum extraction should be terminated when air begins to pass through the filter.

7.4 The extract may be analyzed for conductivity and soluble cations if the 1:1 extract method was not used.

8.0 REFERENCES

8.1 Rhoades, J. D. 1996. Salinity, Electrical Conductivity and Total Dissolved Solids. In D. L. Sparks (ed.) Methods of Soil Analysis. Part 3. Chemical Methods. SSSA Book Series No. 5. ASA Madison, WI.

8.2 U.S. Salinity Laboratory. 1954. L.A. Richards (ed.). Diagnosis and Improvements of Saline and Alkali Soils. U.S Dept. of Agriculture Handbook No. 60.

SOLUBLE CATION EXTRACTION PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 This method applies to most soils and solid wastes. This extract is analyzed for conductivity, calcium, magnesium and sodium. The conductivity is corrected to a conductivity at saturation (see the conductivity section). The cations are used to calculate the sodium adsorption ratio and they are also used to correct for soluble cations in the exchangeable cation procedure.

2.0 INTERFERENCES

2.1 Not applicable.

3.0 APPARATUS

3.1 Filter funnels or centrifuge with the appropriate centrifuge tubes.

3.2 Filter paper.

3.3 Bottles for sample suspensions and filtrates.

4.0 PROCEDURE

4.1 Place at least 100 grams of sample in a bottle and add an equal amount of Type II water.

4.2 Stopper and agitate in a mechanical shaker for 15 minutes. Allow the contents to stand for 12 to 24 hours.

4.3 Agitate again for 5 minutes, and filter or centrifuge and retain the extract or supernatant for analysis.

4.4 Analyze the extract for sodium, magnesium and calcium by atomic absorption spectrophotometry or inductively coupled plasma emission spectroscopy or equivalent EPA approved method. Calculate the sodium adsorption ratio as follows:

$$\text{Na}/\sqrt{(\text{Ca}+\text{Mg})/2}$$

Where:

Na = milliequivalents per liter of sodium

Ca = milliequivalents per liter of calcium

Mg = milliequivalents per liter of magnesium

5.0 REFERENCES

5.1 Rhoades, J. D. 1996. Salinity, Electrical Conductivity and Total Dissolved Solids. In D. L. Sparks (ed.) Methods of Soil Analysis. Part 3. Chemical Methods. SSSA Book Series No. 5. ASA Madison, WI.

5.2 Diagnosis and Improvements of Saline and Alkali Soils. U.S Dept. of Agriculture Handbook No. 60.

OIL AND GREASE EXTRACTION METHOD FOR SLUDGE SAMPLES

1.0 SCOPE AND APPLICATION

1.1 This method is used to recover oil and grease by chemically drying a wet sludge sample and then extracting via the Soxhlet apparatus.

1.2 This method applies only to samples collected for Oil and Grease analysis in accordance with the provisions of LAC 43:XIX.Subpart 1.Chapter 3.

1.3 This method is used when relatively polar, heavy petroleum fractions are present, or when the levels of nonvolatile hydrocarbons challenge the solubility limit of the solvent. This method is not specifically recommended for measurement of low-boiling fractions that volatilize at temperatures below 70 deg. C.

2.0 SUMMARY OF METHOD

2.1 A 25 g sample of wet sludge with a known dry-solids content is used in this procedure.

2.2 Anhydrous sodium sulfate will combine with water and is used to dry the acidified sludge sample. After drying, the oil and grease is extracted with methylene chloride using the Soxhlet apparatus.

3.0 INTERFERENCES

3.1 Caution must be taken to prevent sample particles from falling into the flask containing the solvent.

3.2 The rate and time of extraction in the Soxhlet apparatus must be exactly as directed because of varying solubilities of the different greases.

3.3 The length of time required for drying and cooling extracted material must be constant.

3.4 A gradual increase in weight may result due to the absorption of oxygen; a gradual loss of weight may result due to volatilization.

4.0 APPARATUS AND MATERIALS

4.1 Extraction apparatus: Soxhlet.

4.2 Analytical balance.

4.3 Vacuum pump or some other vacuum source.

4.4 Extraction thimble: Filter paper.

- 4.5 Grease free glass wool or small glass beads to fill thimble.
- 4.6 Beaker: 150 ml.
- 4.7 pH Indicator to determine acidity.
- 4.8 Porcelain mortar.
- 4.9 Extraction flask: 150 ml.
- 4.10 Distilling apparatus: Waterbath at methylene chloride boiling point.
- 4.12 Desiccator.

5.0 REAGENTS

- 5.1 Concentrated hydrochloric acid (HCl).
- 5.2 Anhydrous Sodium sulfate
- 5.3 Methylene Chloride: Boiling point, 40.1 C. The solvent should leave no measurable residue on evaporation; distill if necessary.
- 5.4 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 6.1 Transfers of the solvent methylene chloride should not involve any plastic tubing in the assembly.
- 6.2 Sample transfer implements: Implements are required to transfer portions of solid, semisolid, and liquid wastes from sample containers to laboratory glassware. Liquids may be transferred using a glass hypodermic syringe. Solids may be transferred using a spatula, spoon, or coring device.
- 6.3 Any turbidity or suspended solids in the extraction flask should be removed by filtering through grease-free sodium sulfate.

7.0 PROCEDURE

- 7.1 Weigh out 25g (+/- 0.5 g) of wet sludge with a known dry-solid content, place in a 150 ml beaker.
- 7.2 Acidify to pH of 2 with approximately 0.3 ml concentrated HCl.
- 7.3 Add anhydrous sodium sulfate and stir until the sample appears dry. Spread paste on sides of beaker to facilitate drying. Let stand until substance is solidified.
- 7.4 Quantitatively transfer the sample to the paper extraction thimble.
- 7.5 Fill thimble with glass wool (or glass beads).

7.6 Extract in Soxhlet apparatus using methylene chloride at a rate of 20 cycles/hr for a minimum of 6 hours or until the solvent is colorless.

7.7 Using grease-free glass wool, filter the extract into a pre-weighed 250 ml boiling flask. Use forceps to avoid adding fingerprints to the flask.

7.8 Rinse flask and glass wool with solvent.

7.9 Connect the boiling flask to the distilling head and evaporate the solvent by immersing the lower half of the flask in water at 70 deg. C. Collect the solvent for reuse. A solvent blank should accompany each set of samples.

7.10 When the temperature in the distilling head reaches 50 deg. C. or the flask appears dry, remove the distilling head. To remove solvent vapor, sweep out the flask for 15 seconds with air by inserting a glass tube that is connected to a vacuum source. Immediately remove the flask from the heat source and wipe the outside to remove excess moisture and fingerprints.

7.11 Place the boiling flask in a desiccator until cool and weigh.

7.15 Calculate oil and grease as a percentage of the total dry solids.

$$\% \text{ oil and grease} = 100 * G \div D$$

$$D = (W * 100) \div (100 + (100 * (W - D) \div D))$$

Where: G = Gain in weight of the flask
 D = Dry weight of sample in grams
 W = Wet weight of sample in grams

8.0 QUALITY CONTROL

8.1 Before processing any samples, the analyst should demonstrate through the analysis of a Type II water method blank that all glassware is free of organic contamination; if there is a change in reagents, a method blank should be processed as a safeguard against reagent contamination. The blank sample should be carried through all stages of the sample preparation and measurement.

8.2 Standard quality assurance practices should be used with this method. Laboratory replicates should be analyzed to validate the precision of the analysis. Samples fortified with Wesson oil or its equivalent should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis.

8.3 All quality control data should be maintained and available for easy reference or inspection.

8.4 Employ a minimum of one blank per sample batch to determine if contamination has occurred.

8.5 Run one spike and one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process.

9.0 **REFERENCES**

9.1 Blum, K.A. and M.J. Taras. Determination of Emulsifying Oil in Industrial Wastewater. JWPCF Research Suppl. ,40, R404 (1968).

9.2 Standard Methods for the Examination of Water and Wastewater, 14th. ed., p. 515, Method 502A (1975).

9.3 U.S. Environmental Protection Agency. 1986. Test Methods for Evaluating Solid Wastes. Office of Soil Waste and Emergency Response. USEPA Washington D.C.

pH

1.0 SCOPE AND APPLICATION

1.1 This method is an electrometric procedure which has been approved for measuring pH in calcareous and noncalcareous soils and solid waste.

2.0 SUMMARY OF METHOD

2.1 The dried and prepared sample (soil or solid waste) is mixed with Type II water . The pH of the solution is then measured with a pH meter.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrect pH measurements.

3.2 Temperature fluctuation will cause measurement errors.

3.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can either (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCL so that the lower third of the electrode is submerged, and then thoroughly rinsed with water.

4.0 APPARATUS AND MATERIALS

4.1 pH Meter with means for temperature compensation.

4.2 Electrodes:

4.2.1 Calomel electrode.

4.2.2 Glass electrode.

4.2.3 A combination electrode can be employed instead of calomel or glass.

4.5 Beaker: 50 ml

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Primary standard buffer salts are available from the National Bureau of Standards (NBS) and should be used in situations where extreme accuracy is necessary. Preparation of

reference solutions from these salts require some special precautions and handling, such as low-conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.

5.3 Secondary standard buffers may be prepared from NBS salts or purchased as solutions from commercial vendors. These commercially available solutions, which have been validated by comparison with NBS standards, are recommended for routine use.

6.0 SAMPLE PREPARATION

6.1 See sample preparation section in this manual.

7.0 PROCEDURE

7.1 Calibration

7.1.1 Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

7.1.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value.

7.2 Sample preparation and pH measurement of samples:

7.2.1 To 20 g of sample in a 50-ml beaker, add 20 ml of Type II water and stir the suspension 3 times during the next 30 min.

7.2.2 Let the sampled suspension stand, covered, for 1 hr. to allow most of the suspended clay to settle out from the suspension.

7.2.3 For combination electrodes, immerse just below the suspension. Read the pH to the nearest tenth of a pH unit.

7.2.4 If the sample temperature differs by more than 2 deg. C. from the buffer solution, the measured pH values must be corrected.

7.2.5 Report the results as "pH ,1:1 aqueous."

8.0 **QUALITY CONTROL**

8.1 Duplicate samples and check standards should be analyzed routinely.

8.2 Electrodes must be thoroughly rinsed between samples.

9.0 **REFERENCES**

9.1 EPA METHOD 9045 as amended in the most recent edition of SW-846.

CATION EXCHANGE CAPACITY

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to most soils and solid waste, including calcareous and non-calcareous samples.

2.0 SUMMARY OF METHOD

2.1 The sample is mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added, which replaces the adsorbed sodium with ammonium. The concentration of displaced sodium is then determined by atomic absorption, emission spectroscopy, or an equivalent means as available and approved by EPA.

3.0 INTERFERENCES

3.1 Interferences can occur during analysis of the extract for sodium content. Thoroughly investigate the chosen analytical method for potential interferences.

4.0 APPARATUS AND MATERIALS

4.1 Centrifuge tube and stopper: 50-ml, round-bottom, narrow neck.

4.2 Mechanical shaker.

4.3 Volumetric flask: 100 ml.

5.0 REAGENTS

5.1 Sodium acetate (NaOAc), 1.0 N: Dissolve 136 g of $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ in water and dilute it to 1,000 ml. The pH of this solution should be 8.2. If needed, add a few drops of acetic acid or NaOH solution to bring the reaction of the solution to pH 8.2.

5.2 Ammonium acetate (NH_4OAc), 1 N: Dilute 114 ml of glacial acetic acid (99.5%) with water to volume of approximately 1 liter. Then add 138 ml of concentrated ammonium hydroxide (NH_4OH) and add water to obtain a volume of about 1,980 ml. Check the pH of the resulting solution, add more NH_4OH , as needed, to obtain a pH of 7, and dilute the solution to a volume of 2 liters with water.

5.3 Isopropyl alcohol: 99%

6.0 SAMPLE PREPARATION

6.1 Samples are prepared according to the sample pretreatment section of this manual.

7.0 **PROCEDURE**

7.1 Weigh 5 g of sample to the nearest one hundredth of a gram and transfer the sample to a 50-ml, round-bottom, narrow-neck centrifuge tube.

7.2 Add 30 ml of 1.0 N NaOAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear (free of suspended solids).

7.3 Decant the liquid, and repeat Paragraph 7.2 three more times.

7.4 Add 30 ml of 99% isopropyl alcohol, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

7.5 Repeat the procedure described in Paragraph 7.4 two more times.

7.6 Add 30 ml of NH_4OAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear. Decant the washing into a 100-ml volumetric flask.

7.7 Repeat the procedure described in Paragraph 7.6 two more times.

7.8 Dilute the combined washing to the 100 ml mark with ammonium acetate solution and determine the sodium concentration by atomic absorption, emission spectroscopy, or an equivalent EPA approved method.

8.0 **QUALITY CONTROL**

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 Materials of known cation-exchange capacity must be routinely analyzed.

9.0 **REFERENCES**

9.1 U.S. Environmental Protection Agency. 1986. Methods of Evaluating Solid Wastes. Office of Solid Waste and Emergency Response, Washington D.C.

9.2 This method is based on Chapman, H.D., "Cation-exchange Capacity," pp. 891-900, in C.A. Black (ed.), Method of Soil Analysis, Part 2: Chemical and Microbiological Properties, Am. Soc. Agron., Madison, Wisconsin (1965).

EXCHANGEABLE CATIONS

1.0 SCOPE AND APPLICATIONS

1.1 This method is applicable to most soils and solid wastes and is used to determine the distribution of cations adsorbed on the solid phase.

2.0 SUMMARY OF METHOD

2.1 The sample is mixed with an excess of ammonium acetate solution resulting in an exchange of ammonium with the adsorbed cations. The cations released in the solution are then quantified. The exchangeable cations are then calculated as the difference between the ammonium acetate extracted cations and the soluble cations.

3.0 INTERFERENCES

3.1 Sparingly soluble salts may give erroneously high cation distribution values. Samples that have a high pH may result in inadequate desorption.

4.0 APPARATUS AND MATERIALS

4.1 Centrifuge

4.2 50 ml round-bottom, narrow-neck centrifuge tubes

4.3 Mechanical shaker

4.4 Ammonium acetate solution, 1.0 N.: To 700 or 800 ml of water add 57 ml of concentrated acetic acid and then 68 ml of concentrated ammonium hydroxide. Dilute to a volume of 1 liter and adjust to pH 7.0 by the addition of more ammonium hydroxide or acetic acid.

5.0 SAMPLE PREPARATION

5.1 See the sample preparation section of this manual.

6.0 PROCEDURE

6.1 Weigh 5 grams of sample to the nearest hundredth of a gram and transfer the sample to a 50 ml, round bottom, narrow neck centrifuge tube.

6.2 Add 30 ml. of ammonium acetate reagent to the tube, stopper, and shake it in a mechanical shaker for 5 min and centrifuge it until the supernatant liquid is clear.

6.3 Decant the supernatant liquid as completely as possible into a 100 ml. volumetric flask. Extract with ammonium acetate a total of 3 times by this procedure, decanting into the same flask.

6.4 Dilute to volume, mix, and determine the amounts of the various extracted cations by flame photometric or equivalent EPA approved methods.

6.5 The soluble cations must be subtracted from the exchangeable cations since both are extracted in this procedure. Use the cations measured in the water extract in the calculations below.

7.0 **CALCULATIONS**

Ammonium acetate extractable cations in meq./100 gm = (cation conc. of extract in meq/l X 10) / (wt. of sample in gm).

Soluble cations in meq/100 gm = (cation conc. of saturation extract in meq/l) X (saturation percentage) / 1,000.

Exchangeable cations in meq/100 gm = (extractable cations in meq/100 gm).

8.0 **REFERENCES**

8.1 Chapman, H.D. "Cation-exchange Capacity" pp 891-900, in C.A. Black (ed.), Methods of Soil Analysis, Part 2 : "Chemical and Microbiological Properties, Am. Soc. Agron., Madison, Wisconsin. 1965.

ELECTRICAL CONDUCTIVITY

1.0 SCOPE AND APPLICATION

1.1 This method applies to soils and solid wastes. The electrical conductivity is an indicator of the quantity of soluble salts in a sample.

2.0 SUMMARY OF METHOD

2.1 The conductivity of a 1:1 aqueous extract is measured and the reading is corrected to a conductivity at 25 C at sample saturation. If the conductivity exceeds the regulatory limit the EC is determined on the paste extract.

3.0 INTERFERENCES

3.1 Conductivity varies with temperature, therefore the conductivity must be temperature corrected to 25 C if it is not measured at this temperature.

4.0 APPARATUS AND MATERIALS

- 4.1 Conductivity meter
- 4.2 Conductivity cell
- 4.3 Vacuum filtration device

5.0 REAGENTS

- 5.1 ASTM Type II water
- 5.2 Standard potassium chloride solution

5.2.1 Dissolve 0.7456 g potassium chloride that has been dried in an oven at 105 C overnight in distilled water and bring the volume up to 1 liter for a standard conductance solution of 1.412 millimhos per centimeter at 25 C (commercially available).

6.0 SAMPLE PREPARATION

- 6.1 Prepare the sample according to the sample preparation section in this manual.

7.0 PROCEDURE

7.1 Perform the soluble cation extraction by the method listed in this manual to obtain the solution on which the conductivity will be measured.

- 7.2 Standardize the conductivity meter with the standard potassium chloride by the

manufacturer's directions.

7.3 Rinse the cell with sample solution obtained from the soluble cation extraction procedure. Read the conductivity in millimhos per centimeter and correct the reading to 25 C.

7.4 Use the saturation percentage determined in the saturation paste section of this manual to correct the 1:1 reading to a conductivity at saturation as follows:

$$\text{ELECTRICAL CONDUCTIVITY AT SATURATION} = \text{EC}/\text{S}$$

Where:

EC= THE CONDUCTIVITY MEASURED IN SECTION 7.3 (1:1 AQUEOUS EXTRACT)

S = THE SATURATION PERCENTAGE EXPRESSED AS A DECIMAL

ie: 70 ml required to achieve saturation of 100 grams of sample, $S=.7$
If $\text{EC}=3.0$, then $\text{EC at saturation} = 3.0/.7 = 4.29$ millimho's per cm

7.5 If the EC is above the regulatory limit or S is greater than 1.0, then determine the conductivity on the saturated paste extract.

8.0 QUALITY CONTROL

8.1 Duplicate and check samples must be analyzed routinely.

8.2 A record of the cell constant should be kept to determine when the cell must be replatinized.

9.0 REFERENCES

9.1 Rhoades, J. D. 1996. Salinity, Electrical Conductivity and Total Dissolved Solids. In D. L. Sparks (ed.) Methods of Soil Analysis. Part 3. Chemical Methods. SSSA Book Series No. 5. ASA Madison, WI.

ACID DIGESTION OF SLUDGES

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic adsorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP). Samples prepared by this method may be analyzed by ICP for all the listed metals, or by FLAA or GFAA as indicated below (see also Paragraph 2,1), Mercury is analyzed by the cold vapor technique.

1.1 This method applies only to samples collected for metals analyses in accordance with the provisions of LAC 43:XIX.Subpart 1.Chapter 3.

FLAA	GFAA	HYDRIDE/COLD VAPOR
Barium	Arsenic	Arsenic
Cadmium	Cadmium	Selenium
Chromium	Chromium	Mercury
Lead	Selenium	
Silver		
Zinc		

2.0 SUMMARY OF METHOD

2.1 A representative 1g sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with nitric acid or hydrochloric acid. Dilute hydrochloric acid is used as the final reflux acid for (1) the hydride analysis of As and Se, and (2) the flame AA or ICP analysis of Ba, Ca, Cd, Cr, Pb, and Zn.

3.0 INTERFERENCES

3.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether Method 3050 is applicable to given waste.

3.2 Hydride interferences

3.2.1 High concentrations of chromium, cobalt, copper, mercury, molybdenum, nickel, and silver can cause analytical interferences.

3.2.2 Traces of nitric acid left following the sample work-up can result in analytical interferences. Nitric acid must be distilled off by heating the sample until fumes of SO₃ are observed.

3.2.3 Elemental arsenic and selenium and many of their compounds are volatile;

therefore, certain samples may be subject to losses of arsenic during sample preparation.

4.0 APPARATUS AND MATERIALS

- 4.1 Conical Phillips breakers: 250-ml
- 4.2 Watch glasses
- 4.3 Thermometer: That covers range of 0 to 200 C
- 4.4 Whatman No. 41 filter paper (or equivalent)
- 4.5 Centrifuge and centrifuge tubes

5.0 REAGENTS

- 5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.
- 5.2 Concentrated nitric acid, reagent grade (HNO_3): Acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used.
- 5.3 Concentrated hydrochloric acid, reagent grade (HCl): Acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used.
- 5.4 Hydrogen peroxide (30%) (H_2O_2): Oxidant should be analyzed to determine level of impurities.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 6.1 All samples must have been collected using a sampling plan that results in a representative sample.
- 6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

7.0 PROCEDURE

- 7.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh to the nearest 0.1 mg and transfer to a conical beaker a 1 g portion of sample.
- 7.2 Add 10 ml of 1:1 HNO_3 , mix the slurry, and cover with a watch glass. Heat the sample to 95 deg. C and reflux for 10 to 15 min without boiling. Allow the sample to cool, add 5 ml of concentrated HNO_3 , replace the watch glass, and reflux for 30 min. Repeat this last step to ensure complete oxidation. Using a ribbed watch glass, allow the solution to evaporate to 5 ml without boiling, while maintaining a covering of solution over the bottom of the beaker.
- 7.3 After Step 7.2 has been completed and the sample has cooled, add 2 ml of Type II water and 3 ml of 30% H_2O_2 . Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool

the beaker.

7.4 Continue to add 30% H_2O_2 in 1-ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

NOTE: Do not add more than a total of 10 ml 30% H_2O_2 .

7.5 If the sample is being prepared for (a) the hydride analysis of As and Se, or (b) the flame AA or ICP analysis of Ba, Ca, Cd, Cr, Cu, Pb, and Zn, then add 5 ml of concentrated HCL and 10 ml of Type II water, return the covered beaker to the hot plate, and reflux for an additional 15 min without boiling. After cooling, dilute to 100 ml with Type II water. Particulates in the digestate that may clog the nebulizer should be removed by filtration, by centrifugation, or by allowing the sample to settle.

7.5.1 Filtration: Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 ml with Type II water.

7.5.2 Centrifugation: Centrifugation at 2,000-3,000 rpm for 10 min is usually sufficient to clear the supernatant.

7.5.3 The diluted sample has an approximate acid concentration of 5.0% (v/v) HCL and 5.0% (v/v) HNO_3 . The sample is now ready for analysis.

7.6 If the sample is being prepared for the furnace analysis of As, Cd, Cr, Pb and Se, cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 ml. After cooling, dilute to 100 ml with Type II water. Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle.

7.6.1 Filtration: Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 ml with Type II water.

7.6.2 Centrifugation: Centrifugation at 2,000-3,000 for 10 min is usually sufficient to clear the supernatant.

7.6.3 The diluted digestate solution contains approximately 5% (v/v) HNO_3 . For analysis, withdraw aliquots of appropriate volume and add any required reagent or matrix modifier. The sample is now ready for analysis.

7.7 Calculations:

7.7.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired then the percent solids of the sample must also be provided.

7.7.2 If percent solids is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample.

8.0 QUALITY CONTROL

8.1 For each group of samples processed, preparation blanks (Type II water and reagents) should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

8.2 Duplicate samples should be processed on a routine basis. Duplicate samples will be used to determine precision. The sample load will dictate the frequency, but 20% is recommended.

8.3 Spiked samples or standard reference materials must be employed to determine accuracy. A spiked sample should be included with each group of samples processed and whenever a new sample matrix is being analyzed.

8.4 The concentration of all calibration standards should be verified against a quality control check sample obtained from an outside source.

9.0 METHOD PERFORMANCE

9.1 No data provided.

10.0 REFERENCES

10.1 U.S. Environmental Protection Agency. 1986. Test Methods for Evaluating Solid Waste. Office of Solid Waste and Emergency Response, USEPA, Washington D.C.

TOTAL BARIUM

1.0 SCOPE AND APPLICATIONS

1.1 This method is used to determine the total amount of barium in an Exploration and Production Waste material.

2.0 SUMMARY OF METHOD

2.1 The sample is dried and pulverized to pass a 100 mesh sieve and digested in nitric acid. Atomic absorption or ICP emission spectroscopy is used to determine the barium concentration.

3.0 INTERFERENCES

3.1 Since barite (barium sulfate) is only sparingly soluble in acid, the analyst must strictly adhere to the size of the sample and the volume of acid that is specified in the protocol.

4.0 APPARATUS AND MATERIALS

- 4.1 Erlenmeyer flask, 500 ml
- 4.2 Mortar and Pestle, agate
- 4.3 Nitric acid, 3+7
- 4.4 100 mesh sieve
- 4.5 Volumetric flask, 500 ml

5.0 PROCEDURE

- 5.1 Prepare the sample as in the sample preparation section of this manual.
- 5.2 Grind a 10 gram sample to pass a 100 mesh sieve.
- 5.3 Weigh to the nearest tenth of a milligram 100 mg of prepared sample.
- 5.4 Place the weighed sample into a 500 ml flask and add 400 ml 3+7 nitric acid.
- 5.5 Reflux on a hot plate until the volume is 300 ml.
- 5.6 Transfer to a 500 ml volumetric flask and bring up to the mark.
- 5.7 Determine the barium concentration by atomic absorption or ICP emission spectroscopy.
- 5.8 Report the barium content as parts per million on a dry weight basis.

6.0 REFERENCES

6.1 Lloyd Deuel and B.D. Freeman "Chemical Partitioning of Barium with Soil at Various Redox Potentials", Soil Science Society American Journal, submitted 1988.

LEACHATE TEST

1.0 SCOPE AND APPLICATION

1.1 This method is designed to simulate water leach effects on treated Exploration and Production Wastes addressed specifically under LAC 43:XIX.Subpart 1.Chapter 3 and LAC 43:XIX.565.F.

2.0 SUMMARY OF METHOD

2.1 A representative sample of treated waste is extracted by continuous contact with water at a 1:4 solid:solution ratio. Samples are reacted for 7 days at 25 C +/- 2 C with flask contents swirled on a daily basis to effect gentle, intermittent agitation. A suitable aliquot of the extract is analyzed for chlorides with the remainder of the sample partitioned for oil and grease by separatory funnel liquid-liquid extraction using methylene chloride (LAC 43:XIX.Subpart 1.Chapter 3) or total petroleum hydrocarbons by EPA Method SW-846 1664/SGT / SW-846 9071 (LAC 43:XIX.565.F).

2.2 Soil separates generated from land treatment of Exploration and Production Waste materials are extracted as received. Solidified Exploration and Production Wastes are fractured to pass a 9.5 mm sieve prior to extraction.

3.0 INTERFERENCES

3.1 Refer to applicable analytical methods for chlorides, oil and grease and total petroleum hydrocarbons for discussion of potential interferences that may be encountered during analysis. Naturally occurring organic matter (i.e. humic acids) may be co-extracted and impart a positive error to the Exploration and Production Waste leach test. Gas chromatographic analysis may be used to differentiate naturally occurring organic matter from Exploration and Production Waste products partitioned into the water extract.

4.0 APPARATUS AND MATERIALS

4.1 Extraction apparatus: 1000 ml erlenmeyer flask, rubber stoppers.

4.2 Analytical balance, weighing beakers, desiccator.

4.3 Separatory funnel and filter apparatus.

5.0 REAGENTS

5.1 ASTM Type II water

5.2 Nanograde methylene chloride

5.3 Reagent grade anhydrous sodium sulfate

6.0 SAMPLE PREPARATION

6.1 Land treatment or otherwise non-solidified solids are used as received, following careful mixing to obtain a representative subsample.

6.2 Sample moisture level is determined on a separate subsample to correct for interstitial water in preparing the 1:4 solid:solution test matrix.

6.3 Solidified materials are fractured to pass a 9.5 mm sieve. Moisture determinations are made for representative aggregate to correct for interstitial water in preparing the 1:4 solid:solution test matrix.

7.0 PROCEDURE

7.1 Weigh 100 grams (dry weight basis) prepared sample into a 1000 ml extraction flask.

7.2 Add Type II water as required to adjust solution volume to 400 ml. Stopper and swirl contents of flask to initiate 7 day leach test.

7.3 Contents are swirled on a daily basis to effect gentle agitation of the sample matrix.

7.4 The sample extract is separated from solids by filtration. An aliquot of the filtrate is analyzed for chloride using EPA Method SW-846 9056/9253.

7.5 For sample analysis under LAC 43:XIX.Subpart 1.Chapter 3, oil and grease is partitioned from the filtrate using methylene chloride (EPA Method 3510). The procedure entails three extractions with 60 ml of methylene chloride. Extracts are combined, dried over solvent rinsed anhydrous sodium sulfate and reduced in volume for quantitative transfer to a preweighed beaker. The sample extract is finally dried in a desiccator for gravimetric analysis of the oil and grease component. For sample analysis under LAC 43:XIX.565.F, use procedures detailed in EPA Method SW-846 1664 HEM/SGT / SW-846 9071.

7.6 Calculate and report test parameter results in mg/l.

8.0 QUALITY CONTROL

8.1 Comprehensive quality control procedures are specified for chlorides, oil and grease and total petroleum hydrocarbons by the referring to applicable analytical methods.

8.2 All quality control data should be maintained and available for easy reference and inspection. Data should include reagent blank, duplicate and spike recovery analysis.

9.0 REFERENCES

9.1 Texas Water Commission, Industrial Solid Waste Management, Waste Evaluation/Classification, Technical Guide #1.

LAND TREATMENT CELL SAMPLING PROCEDURES

1.0 SCOPE AND APPLICATION

1.1 The procedures outlined herein may be used as guidance for obtaining representative samples from land treatment cells for TCLP Benzene analysis of soils pursuant to Testing Criteria for Reusable Material (LAC 43:XIX.565.F). It is incumbent upon land treatment facility operators to assure that representative samples are properly obtained, preserved and shipped to laboratories for applicable analytical analyses.

2.0 LAND TREATMENT CELL SAMPLING PROCEDURE

2.1 Soil samples in land treatment cells shall be taken in the waste treatment zone (WTZ) and the upper treatment zone (UTZ).

2.2 At least two randomly selected, representative samples must be taken from the WTZ and the UTZ for each acre of cell area.

2.3 All samples from the WTZ and the UTZ may be composited for one representative analysis for each zone from each cell.

3.0 SOIL / SLUDGE MATRIX SAMPLING PROCEDURE

3.1 If the matrix is predominately solids with little or no free liquids present, the following technique should be used:

3.1.1 The sampler shall acquire a representative solid sample using the best available sampling tool (core sampler, etc.) and the core sample obtained shall be placed immediately into a clean, 2-ounce wide-mouth glass sampling jar with a sealable, air-tight Teflon lined cap.

3.1.2 The sample jar shall be filled as full as possible to avoid air space above the solid sample, and the sample jar should be capped immediately after filling.

3.1.3 The sample jar shall be properly labeled and placed on ice immediately after sampling.

4.0 LIQUID / SLURRY MATRIX SAMPLING PROCEDURE

4.1 If the matrix is predominately free liquid or a slurry, the following technique should be used:

4.1.1 The sampler shall acquire a representative liquid /slurry sample using the best available sampling tool(s), and the sample shall be placed in a clean, unpreserved 16 ounce wide-mouth glass sampling bottle with a sealable, air-tight Teflon lined cap.

4.1.2 The sample bottle shall be filled completely to avoid any air bubbles at the top of the bottle, then immediately capped.

4.1.3 The sample bottle shall be labeled and placed on ice immediately after sampling.

5.0 SAMPLE COMPOSITING

5.1 All sample compositing performed under these procedures for TCLP Benzene must be performed at the receiving laboratory facilities.

5.1.1 Upon sample collection and prior to shipment, the sample jars / bottles must be clearly labeled.

5.1.2 Samples must be shipped with a properly completed chain of custody clearly identifying which samples are to be composited in accordance with the sampling plan.

5.1.3 Following completion of sample compositing, the analytical laboratory will proceed with the required testing.

6.0 GENERAL QUALITY CONTROL

6.1 The sampler should be prepared to sample a variety of matrices that may be present at the land treatment cell facility. It is advised that the sampler contact the laboratory to discuss the sampling plan prior to the sampling event.

6.1.2 The proper number of sample containers, container types, preservatives, chain of custodies, etc. should be acquired from the laboratory prior to the sampling event.

6.1.3 All appropriate field equipment cleaning techniques and requirements shall be utilized during field sampling.

7.0 MISCELLANEOUS

7.1 The Land Treatment Cell Sampling Procedure outlined in section 2.0 may be utilized to sample for criteria other than TCLP Benzene pursuant to LAC 43:XIX.565.F, Testing Criteria for Reusable Material.

7.2 Sample compositing may be performed in the field for criteria other than TCLP Benzene.

APPENDICES

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