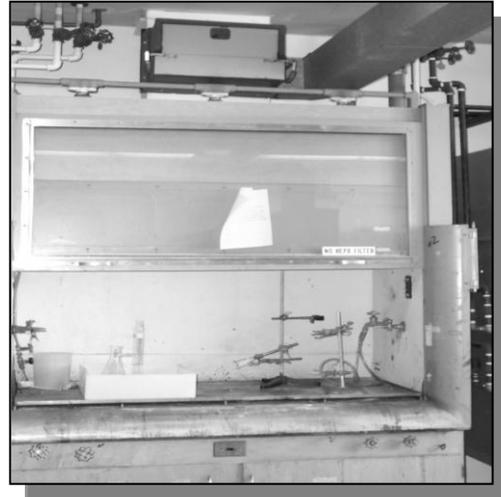


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# Perchlorate and Perchloric Acid Sampling and Analysis Procedure

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## 1.0 Purpose/Scope

This document describes a field procedure for taking and analyzing wipe samples of surfaces potentially contaminated with perchlorates from perchloric acid use. It is based on methodology described in *Perchloric Acid Contaminated Hood Decontamination Procedure Manual* (1993), prepared by ORNL. The goal of the procedure is to provide a uniform method to collect representative samples of surface contamination (such as lab hoods and duct work) and to provide a standardized, accurate method to analyze the concentration. Using this method will ensure repeatability between various sampling personnel and surface configurations.

This field procedure describes practices developed from lessons learned experience. This procedure should be viewed as a best management practice. Regulatory limits for surface contamination with perchlorates do not exist, but this method allows quantification of surface levels for comparison with known safe levels from industry experience.

## 2.0 Responsibilities

- 2.1 This program is implemented through the SHSD Industrial Hygiene Manager who may delegate authority to administer this program. Personnel who have demonstrated competency in performing a certain role, in accordance with Section 7 of this procedure, will be qualified to serve that role.

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- 2.2 Chain of Custody procedures: The collector of the sample is responsible for the integrity of the sample until the sample has been properly transferred to the IH Group laboratory. It is permissible to use this procedure to collect samples that will be analyzed by a laboratory not associated with the SHSD IH Group.
- 2.3 Hazard Analysis of the Sampling Task: It is the responsibility of the person using this method and his/her supervisor to ensure that the appropriate personal protective equipment is worn while performing this procedure. In addition, the person performing this procedure and his/her supervisor are responsible to ensure that all required training and qualification for hazards that may be present in areas where this procedure will be used (such as respiratory protection or radiation contamination) have been met. The person performing this procedure and his/her line supervisor are responsible to comply with all work planning and work permit system requirements.

### 3.0 Definitions

**Perchlorate(s):** Compound containing a monovalent  $\text{ClO}_4^-$  radical, including salts of perchloric acid, such as Ammonium perchlorate and magnesium perchlorate. Unstable material, flammable by chemical reaction, powerful oxidizer, explosive hazard when shocked, exposed to heat, or chemical reaction.

**Perchloric Acid:** Colorless, fuming, unstable liquid, severe explosion hazard,  $\text{ClH}_4\text{O}_4$ .

**Qualified Sampler:** A person who has demonstrated competency to perform field sampling.

**Qualified Analyzer:** A person who has demonstrated competency to perform lab analysis.

### 4.0 Prerequisites

#### 4.1 Training prior to using this procedure:

- 4.1.1 Demonstration of proper operation of the procedure per Section 7 for qualification requirements.
- 4.1.2 Training for hazards other than noise may be needed for entry into restricted areas (check with ESH Coordinator or FS Representative for the facility).

#### 4.2 Area Access:

- 4.2.1 Contact the appropriate Facility Support Representative or Technician to obtain approval to enter radiological areas.
- 4.2.2 Verify with the appropriate Facility Support Representative or Technician if a Work Permit or Radiological Work Permit is needed or is in effect. If so, review and sign the permit.
- 4.2.3 Use appropriate PPE for area.

### 5.0 Precautions

#### 5.1 Personal Protective Equipment:

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**During Sampling:** When sampling interiors of lab hoods, use the sash as a barrier between the sampler and surface being tested to the greatest extent possible. Use Ballistic gear (riot helmet, face shield, and flack jacket) in duct sampling, when the sampling will result in a shock hazard to the duct work.

**Hand:** Use disposable gloves when contacting the surface material and handling exposed sampling media. The gloves must have sufficient impermeability to the surface contaminant and solvent used on the collection media to allow safe handling. Recommended gloves are disposable Nitrile, Natural Latex Rubber, or PVC.



**Body:** If contact of the body with contaminated surfaces is anticipated, a disposable suit should be used. Acceptable chemical protective equipment materials include: Tyvek®, KleenGuard®, and cotton. If personal clothing items become contaminated, they must be surrendered for BNL cleaning or disposal.

**Foot:** If contact of the feet with contaminated surfaces is anticipated, disposable shoe coverings, boots or booties should be used. Acceptable CPC material include: Tyvek®, KleenGuard®, and rubber. If personal shoes become contaminated, they must be surrendered for BNL cleaning or disposal.

**Respiratory:** Under normal use, respiratory protection is not required.

**Eye:** Safety Glasses with side shields are required.

**During Analysis:** Disposable gloves must be used when mixing the 70% Perchloric acid and should be used when handling the glassware and pipettes containing diluted perchlorates. Recommended gloves are disposable Nitrile, Natural Latex Rubber, or PVC. Use safety glasses and a lab coat. Be sure the perchloric acid does not contact organic material.

**Radiation Contamination:** It is possible that some surfaces to be tested may have radiation contamination as well as the chemical contamination. In these cases, personal protective equipment and administrative controls must be implemented for the radiological contaminant hazard in addition to the chemical hazard. In addition, the collected sample must be analyzed for the radiological hazard, where appropriate, before it can be submitted to the IH Group for analysis. At no time will the IH Group accept a sample with radiological contamination above permissible limits for the general public.

**Perchlorate Hazard:** Crystals of perchlorates, when dry, pose an explosion hazard when disturbed. The bottle containing the 70% solution of Perchloric Acid should be kept moist at all times to prevent the formation of perchlorates at the interface of the bottle and cap. To achieve this, the acid bottle is stored within a second bottle containing a moist paper towel.



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**Emergency:** For small spills of perchloric acid/ perchlorate solution, squeegee spilled liquid into a pan and sinks dispose with large volumes of water; use wet paper towels to clean all affected surfaces. Rinse paper towels thoroughly in large volumes of water.

**Work Planning:** All requirements of work permits and work planning system reviews must be met in performing this procedure. A work permit is required for the transport and use of chemicals when SHSD IH Group does analytical work at another facility outside of the SHSD IH Lab.

**Environmental Impact and Waste Disposal:** This technique has minimal adverse impact on the environment. The sampling media used in this technique is processed by the laboratory performing analysis. The process is described in Attachment 9.3 SHSD Operation Environmental Evaluation.

- Ammonium sulfate/perchlorate solutions are sink disposed with copious amounts of water for safety reasons. As per “Liquid Effluent Evaluation Form” dated 08/30/04, if analysis of field samples are conducted in a period when the BNL Sanitary treatment Plant is experiencing elevated levels of Perchlorates, permission for sink release will be suspended and the samples will need to be containerized for waste disposal.
- PPE used during testing or analysis, washing the outer surfaces over a sanitary sink is appropriate. The PPE can then be disposed of in the non-hazardous trash.

**Job Risk Assessment:** Consult the *Job Risk Assessment* [SHSD-JRA-04](#) for the risk analysis of this operation based on the hazards and controls of this SOP.

## 6.0 Procedure

### Sampling Equipment

- 4x4 inch cotton gauze or equivalent
- Rod or extension device
- Distilled water
- Sample containers
- Rubber bands (to hold gauze or pad to extension device)

### Analysis Equipment

- Ion selective electrode
- Water sprayer
- Lab Glassware: Flasks, graduated cylinders, bottles, pipettes, pipettor
- Oakton ION 6 Acorn Series pH/ION/°C meter
- Cole Parmer K-27504-24 Perchlorate probe or equivalent

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## A. Perchlorate Field Sample Collection

### 6.1 Select appropriate sample locations, including assessable points in hoods, exhaust stacks, and fan housings.

From inside the hood, sample can be taken from:

- Walls of the hood, especially near the back by air flow intakes
- Work surface/Counter top
- Within the upper baffle opening
- Within the lower baffle opening
- Around the mouth of the duct, if accessible.

From the exterior of the system, consider sampling:

- Inside of the duct walls, if openings exists (such as isolation dampers, traverse sampling ports, etc.)
- Around the mouth of the duct at the rain cap.
- Fan Housings (via isolation dampers, service panels, etc.)

### 6.2 Sampling a hood, duct, or exhaust stack with a gauze wipe

- Secure a piece of gauze to a wooden rod or other appropriate extension device. A rubber band typically secures the gauzes well.
- Moisten the gauze with the distilled water. Gently dab and hold the wetted gauze to surface to be tested. Move over the surface in a dabbing/holding motion. Avoid hard rubbing and shock to surfaces.
- Using gloves, remove the gauze and place into a labeled sample container.
- Add **25ml** of distilled water.
- Shake the vial for at least one minute.

### 6.3 Sampling a fan housing or duct with liquid wash down

- Spray about 1 liter of distilled water into the fan housing or exhaust opening (system must be off).
- Direct the spray toward the fan while someone slowly turns the fan shaft.
- Allow the water to accumulate at the bottom of the fan housing (or in hood as appropriate).
- Feed Tygon® tubing into the fan housing until the tube reaches the wash water.
- Turn on a suction pump to collect the wash down water. It will provide a sample liquid (sufficient sample volume is 50ml).

### 6.4 Record sampling information: Label the container used to collect the sample and complete a Field Survey Form. The label and form should document the following: Date; Building and room identification; Source identification; Location in the exhaust ventilation system where sampled (i.e., hood, fan, stack, etc.); Suspected sample contents—contaminant;

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Specific location of each sample; Surface Area sampled- estimate (quantify the area, such as 100 cm<sup>2</sup> or 1 square foot.); Samplers name

## B. Sample Analysis for Perchlorates

This method is applicable to aqueous solutions containing perchloric acid and its salts in the range of about 1 to nearly 100,000 ppm perchlorate. It is based on potentiometric measurements of the Electromotive Force (EMF) of a cell composed of an electrode that responds with high specificity to perchlorate ion, and a reference electrode in a solution that is adjusted to 0.2M with ammonium sulfate, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, a high input impedance voltmeter with a sensitivity of 0.1 mV is used to measure the EMF of the cell. The EMF changes by approximately 57 ± 1 mV per decade change in perchlorate concentration.

### 6.5 Ammonium Sulfate solution preparation

- Tare a piece of weighing paper on the balance.
- Weight **13.2 +/- 0.5 grams** of Ammonium Sulfate on the balance.
- Dissolve the 13.2g of Ammonium Sulfate (99+% purity) in approximately 25ml distilled water in a 100 ml beaker.
- Transfer the solution to a 50ml volumetric flask.
- Repeatedly rinse the transfer beaker with distilled water; transfer the rinses to the volumetric flask.
- Bring flask up to the 50 ml mark with distilled water.
- Stopper the flask and invert several times to mix.
- Label flask as "**2M Ammonium Sulfate**" with the current date.



### 6.6 Ion Strength Adjuster (ISA) preparation

- Pipette **10ml** of the 2M Ammonium Sulfate Solution into a **100ml** volumetric flask and fill to the mark with distilled water.
- Label the flask "**0.2M Ion Strength Adjuster**". This solution will be used to minimize the ionic strength error which becomes prevalent at higher concentrations of ions in solution.

### 6.7 Instrument set-up

- **Battery Check:** On the Oakton ION 6 meter, check the battery status by turning the meter "on" (press the "On/Off" button). Replace the AAA batteries if no display occurs or a "LO" annunciator appears on the display.
- **Specific Ion (Perchlorate) Electrode (New or long term storage electrode):** Check the date of manufacture on the probe lead to ensure it is within one year of the current date. Remove and retain the rubber end-cap and slide the rubber insert covering the reference solution filling hole down one inch. Take the Cole Parmer 0.1M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> reference solution from the aqueous liquid



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secondary containment and check that it has not expired. Fill the electrode with the reference solution. Gently shake the electrode to remove any air bubbles which may be trapped behind the membrane. Replace rubber insert over reference solution filling hole. Immerse the probe in deionized water for 30 minutes. Connect the BNC connector from the probe to the meter.

- **Specific Ion (Perchlorate) Electrode (short term storage electrode):** Check that the probe has been stored for less than 30 days in the 10 ppm solution by the date on the beaker and that the solution level has not dropped below the membrane. Remove electrode from 10 ppm storage solution and rinse with deionized water. Connect the BNC connector from the probe to the meter.

#### 6.8 Perchlorate Stock solution preparation

- Pipette **0.43ml** of 70% Perchloric acid into a **500ml** volumetric flask.
- Add distilled water to the 500ml mark, cap the flask, and invert to mix.
- Label as “1000 ppm Perchlorate Stock” and mark the date on the flask.

#### 6.9 Calibration Standards preparation

- Calibration Solution 1: Using a volumetric pipette, pipette 2ml of 0.2M Ammonium Sulfate (ISA) into a 100ml volumetric flask and fill to the mark with distilled water. Label as “0 ppm-perchlorate calibration standard.”
- Calibration Solution 2: Using a volumetric pipette, pipette 0.1ml of the 1000 ppm-perchlorate stock into a 100ml volumetric flask. Add 2ml of 0.2M Ammonium Sulfate (ISA) and fill to the mark with distilled water. Label as “1 ppm-perchlorate calibration standard.”
- Calibration Solution 3: Using a volumetric pipette, pipette 1.0ml of the 1000 ppm-perchlorate stock into a 100ml volumetric flask. Add 2ml of 0.2M Ammonium Sulfate (ISA) and fill to the mark with distilled water. Label as “10 ppm-perchlorate calibration standard.”
- Calibration Solution 4: Using a volumetric pipette, pipette 10.0ml of the 1000 ppm-perchlorate stock into a 100ml volumetric flask. Add 2ml of 0.2M Ammonium Sulfate (ISA) and fill to the mark with distilled water. Label as “100 ppm-perchlorate calibration standard.”

Final Concentration [ppm]	Volume of “1000 ppm Stock”	Volume of Distilled Water	Volume of 0.2M ISA	Total Volume
<b>0</b>	0 ml	98 ml	2.0 ml	100 ml
<b>1</b>	0.1 ml	97.9 ml	2.0 ml	100 ml
<b>10</b>	1.0 ml	97 ml	2.0 ml	100 ml
<b>100</b>	10.0 ml	88 ml	2.0 ml	100 ml

#### 6.10 Calibration Curve analysis (Decade Slope)

- Prepare and label four clean 100 – 200 ml beakers for the calibration solutions.
- Pour 50 ml of each calibration solution into a separate labeled beaker for analysis.

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- Analyze the blank solution first and at the end of each sample set.
- Press the **“On”** button once to turn on the meter.
- Press the **“Mode”** button once to enter the *ppm* mode. Check to ensure that *ppm* mode is active by confirming that no units are displayed in the lower right corner of the display.
- Press the **“Cal”** button to display “0.1”.
- Press the **“Up”** button once to display “1.0”.
- Press **“Enter”** and place the probe tip into the 1 ppm standard solution at a depth of 1 to 2 inches taking care to make sure the membrane is not in contact with the bottom. The black Orion stand is used to position the probe accurately.
- Place a small magnetic stirring bar in the beaker and begin stirring on low speed. Take care not to damage the probe with the stir bar.
- Allow the electrode reading to stabilize. Record the mV value on the *Perchlorate Analysis Record*.
- Press **“Enter”**.
- Shut off the stirring bar and remove the probe from the 1.0 ppm standard perchlorate solution.
- Rinse the probe with deionized water into a separate wash water beaker.
- Confirm that the value “10.0” is flashing in the display.
- Press **“Enter”** and place the probe tip into the 10 ppm standard solution at a depth of 1 to 2 inches taking care to make sure the membrane is not in contact with the bottom.
- Place a magnetic stirring bar in the beaker and begin stirring on low speed. Take care not to damage the probe with the stir bar.
- Allow the electrode reading to stabilize. Record the mV value on the *Perchlorate Analysis Record*.
- Press **“Enter”**.
- Shut off the stirring bar and remove the probe from the 10 ppm standard perchlorate solution.
- Rinse the probe with deionized water into a separate wash water beaker.
- Confirm that the value “100.0” is flashing in the display.
- Press **“Enter”** and place the probe tip into the 100 ppm standard solution at a depth of 1 to 2 inches taking care to make sure the membrane is not in contact with the bottom.
- Place a magnetic stirring bar in the beaker and begin stirring on low speed. Take care not to damage the probe with the stir bar.
- Allow the electrode reading to stabilize. Record the mV value on the *Perchlorate Analysis Record*.
- Press **“Enter”**. Immediately record the Decade Slope value displayed as “PXX”, where XX is a two digit number. This value must be within the probe operating range of 52-59 to continue.
- Shut off the stirring bar and remove the probe from the 100 ppm standard perchlorate solution.



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- Rinse the probe with deionized water into a separate waste water beaker.
- Repeat calibration after every 10-12 field samples and after every two hours of analytical time.

#### 6.11 Sample Analysis

- Pipette 5ml of sample solution from Step 6.2 or 6.3 into a 50ml volumetric flask.
- Add 2ml of 0.2M Ammonium Sulfate (ISA) solution.
- Fill to the 50ml volume mark with distilled water and shake or swirl to mix.
- Transfer to a clean, labeled 100 – 200 ml beaker. Add a magnetic stir bar and immerse electrode probe into the solution. Slowly spin the bar.
- Allow the electrode reading to stabilize. Record the mV and ppm reading on the *Perchlorate Analysis Record*.
- Rinse the electrode with distilled water into a separate wash water beaker between samples and pat dry to prevent cross contamination.
- Repeat steps 6.11 for each sample.
- After analyzing field samples, place the probe into distilled water and record the mV reading in the *Distilled Water Post-Test* line on the *Perchlorate Analysis Record*.
- After analyzing field samples, place the probe into each standard and record the mV and ppm readings in the *Post-Test* lines on the *Perchlorate Analysis Record*.
- Rinse the electrode with distilled water and pat dry between standards to prevent cross contamination.

#### 6.12 Evaluation of Risk: Evaluate the concentration of perchlorates in the field samples by comparison to the calibration solution.

- **Samples with a concentration of <100 ppm in the test solution are considered negative for the risk of a fire or explosion hazard, i.e. no perchlorates.**
- **Samples with a concentration of 100-500 ppm in the test solution are considered suspect and the risk should be based on the results from additional samples taken from the same hood/ ventilation system.**
- **Samples with concentrations >500 ppm in the test solution are considered positive for the presence of perchlorates.**
- If desired, quantify the perchlorate concentration per unit area sampled: Divide the concentration measured (in ppm) by 4 to obtain the total weight of perchlorates in the sample in milligrams. Divide the total weight of perchlorates by the surface area sampled to obtain the total weight per square foot. If the weight of perchlorates is less than 1.25 mg per square foot (equivalent to 100 ppm), then the sample is considered negative for the presence of perchlorates.

#### 6.13 Clean-up of Lab Equipment

- Dilute reagents and field sample solutions with tap water and discharge to sink.
- Flush the sink with water for 3 minutes.
- Wash glassware with detergent. Rinse glassware thoroughly with tap water. Triple rinse with distilled water. Air dry all glassware on racks or countertop.

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- Thoroughly wipe up any spilled perchloric acid on the counter top and equipment with a **wet** paper towel. Thoroughly rinse and wring out the paper towel in the sink three times. Dispose of the paper towel in trash.
- Rinse the outsides of the electrodes in distilled water. If additional analysis is scheduled within 2 weeks, store the electrodes in 10 ppm standard, covered in a manner to prevent evaporation of the water with a label indicating the date placed in storage. Otherwise, rinse the probe membrane with distilled water, pat dry and cover the tip with the black protective cap.
- Drain the reference portion of the combination electrode through the filling hole and replace the rubber insert. Place the electrode in the storage box.

## 7.0 Implementation and Training

Persons who have demonstrated competency in this test to the satisfaction of the SHSD IH Manager or designee will be considered qualified. Personnel will be re-qualified at a frequency not to exceed three years. Attachment 9.3 *Job Performance Measure Completion Certificate* is used to document qualification. Completion of qualification is tracked in the BTMS system.

## 8.0 References

- *Perchloric Acid Contaminated Hood Decontamination Procedure Manual*, Oak Ridge National Laboratory, Oak Ridge, TN, 1993.
- Oakton Ion 6 Acorn Series Operating instructions, Pub # 00702-79, Aug 1999
- Cole Parmer Ion Sensitive Electrodes (ISE) Quick Start Instructions, Rev. 8/11

## 9.0 Attachments

- 9.1 Sample of *Perchlorate Sampling & Analysis Record*
- 9.2 Qualification: Job Performance Measure
- 9.3 Environmental Evaluation

## 10.0 Documentation

<b>Development</b>		
Prepared By: <b>A. Sells &amp; R. Selvey 03/01/00</b>	Reviewed By: <b>R. Selvey 09/27/01</b>	Approved By: <b>R. Selvey 09/28/01</b>
ESH Coordinator/ Date: <i>none</i>	Work Coordinator/ Date: <i>none</i>	SHSD Manager / Date: <i>none</i>
QA Representative / Date: <i>none</i>	Training Coordinator / Date: <i>none</i>	Safety & Health Representative: <i>none</i>
Facility Support Rep. / Date: <i>none</i>	Environ. Compliance Rep. / Date: <i>none</i>	Other: <i>none</i>
ISM Hazard Categorization <input checked="" type="checkbox"/> High <input type="checkbox"/> Moderate <input type="checkbox"/> Low/Skill of the craft	Validation: Formal Walkthrough 07/20/09	Implementation: Document Control:07/21/09 JRA: Reviewed 07/10/12

<b>Revision Log</b>
Rev0: 09/28/01 New procedure published.

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Rev1: Clarify/enhance procedural controls. Revise format in Section 7. Add ballistic gear to Section5. Changes to field sampling in Section 6 including better description of sampling locations. Addition of date and name to Attachment 9.1. Reviewer/Date: 03/12/03 Robert Selvey
Rev2: Clarify/enhance procedural controls. Minor changes to Section 7. Minor format changes to Equipment section. Reviewer/Date: R. Selvey 02/01/06
Rev3: Clarify/enhance procedural controls. Revised Section 4.1 and 5.1 Added Section 4.2.; 5.5, 5.6 and 7.3. Revised Attachment 9.1 and 9.2 with document control (added Revision Date) and 9.3 with Attachment#. Reviewer/Date: R. Selvey 05/23/07
Rev4: Clarify/enhance procedural controls. Added Attachment 9.4. Reviewer/Date: R. Selvey 02/06/09
Rev5: Clarify/enhance procedural controls. Updated Sections 5.3 and 5.4. Corrected step numbering in Section 6. Updated quantities of analytes/reagents and concentration of standards in Section 6. Revised form 9.1. Reviewer/Date: W. Litzke 07/21/09 and A. Meier 07/21/09
Rev6: Clarify/enhance procedural controls. Updated Section 6 for Oakton Ion 6 meter and Cole Parmer ISE probe. Reduced quantities of standards and reagents in Section 6 to reduce waste. Revised Section 8. Revised attachment 9.1. Reviewer/Date: W. Czekaj

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## Attachment 9.1 Sample of Perchlorate Sampling & Analysis Record

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<b>Perchlorate Sampling and Analysis Record</b>	

IH SERVICE NUMBER: _____ Date: _____ Sampler: _____ Analysts: _____ Building No: _____	<table border="1" style="width: 100%;"> <thead> <tr> <th style="text-align: center;">STANDARDS</th> <th style="text-align: center;">Meter Reading (mV)</th> <th style="text-align: center;">Perchlorate Concentration (ppm)</th> </tr> </thead> <tbody> <tr> <td>Blank/Zero (pre-test) 0 ppm</td> <td></td> <td></td> </tr> <tr> <td>Standard 1 ppm</td> <td></td> <td></td> </tr> <tr> <td>Standard 10 ppm</td> <td></td> <td></td> </tr> <tr> <td>Standard 100 ppm</td> <td></td> <td></td> </tr> <tr> <td>Calculated Decade Slope</td> <td></td> <td></td> </tr> </tbody> </table>	STANDARDS	Meter Reading (mV)	Perchlorate Concentration (ppm)	Blank/Zero (pre-test) 0 ppm			Standard 1 ppm			Standard 10 ppm			Standard 100 ppm			Calculated Decade Slope		
STANDARDS	Meter Reading (mV)	Perchlorate Concentration (ppm)																	
Blank/Zero (pre-test) 0 ppm																			
Standard 1 ppm																			
Standard 10 ppm																			
Standard 100 ppm																			
Calculated Decade Slope																			

Perchlorate Concentration < 500 ppm

Results <100ppm are negative; Results >500ppm are positive  
 Results >100ppm but <500ppm are suspect and must be confirmed

#	Room No:	Hood No:	Location or Description	Meter Reading (mV)	Perchlorate Concentration (ppm)	Results Neg/Suspect/Pos
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						

Standard 1 ppm	N/A	
Standard 10 ppm	N/A	
Standard 100 ppm	N/A	
Standard 1000 ppm	N/A	

13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						

Blank/Zero (post-test) 0 ppm		
Standard 1 ppm		
Standard 10 ppm		
Standard 100 ppm		

Analytical Equipment: Oakton Ion 6+ with Cole Parmer K-27504-24 Perchlorate Probe, 25 deg. C

## Perchlorate and Perchloric Acid Sampling & Analysis Job Performance Measure (JPM) Completion Certificate

<b>Candidate's Name</b>	<b>Life Number:</b>
<b>Qualification for:</b> <input type="checkbox"/> <b>Field Sampling Only</b> <input type="checkbox"/> <b>Lab Analysis Only</b> <input type="checkbox"/> <b>Both Field and Lab work</b>	

**Field Sampling**

Criteria	Qualifying Performance Standard	Unsatis- factory	Reco- vered	Satis- factory
<b>1. Hazard Analysis</b>	Understands the need to perform a hazard analysis of the area and potential exposure to the self as sampler and workers in the area.			
<b>2. PPE</b>	Understands the potential for surface contamination, airborne levels of contaminants, radiological hazards, and noise hazard. Knows how to determine the proper PPE.			
<b>3. Sampling Equipment</b>	Knows where equipment needed for the procedure is located and how to properly sign it out.			
<b>4. Measurement of hazard</b>	Knows how to properly take a sample to avoid an uncontrolled reaction and injury.			
	Knows the technique and hood locations to lift the highest possible percentage of Perchlorates from the surface sampled.			
<b>5. Documentation</b>	Demonstrates correctly filling out IH forms, transfers appropriate info to IH databases, and prepares an evaluation assessment report.			

**Lab Analysis**

Criteria	Qualifying Performance Standard	Unsatis- factory	Reco- vered	Satis- factory
<b>1. Hazard Analysis</b>	Understands the hazard of the Perchloric acid stored to make standards & used in the analysis and potential exposure to the self as sampler and workers in the area and the explosion hazard.			
<b>2. PPE</b>	Understands the need to be aware of the potential surface contamination and radiological hazards on submitted samples. Knows the proper PPE.			
<b>3. Sampling Equipment</b>	Knows where equipment needed for the procedure is located and how to properly operate it.			
<b>4. Calibration</b>	Demonstrates the proper set up of the calibration curve with standards.			
<b>5. Measurement of Perchlorates in samples</b>	Knows how to properly prepare field samples with appropriate solutions.			
	Knows how to properly measure field samples with the conductivity meter.			
	Knows how to properly determine the concentration of Perchlorates in the samples by comparison to the calibration curve.			
<b>6. Documentation</b>	Demonstrates correctly filling out IH forms, transfers appropriate info to IH databases, and prepares an evaluation assessment report.			

I accept the responsibility for performing this task as demonstrated within this JPM and the corresponding SOP.

<b>Candidate Signature:</b>	<b>Date:</b>
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I certify the candidate has satisfactorily performed each of the above listed steps and is capable of performing the task unsupervised.

<b>Evaluator Signature:</b>	<b>Date:</b>
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## SHSD Environmental Evaluation of Perchlorate Testing

<p><b>Operation Description:</b> 0- 2000 ppm calibration solutions of Perchloric Acid in Ammonium Sulfate solution are made. Field samples with potential Perchlorates (in distilled water) are tested with a specific ion conductivity probe. The meter response for samples is compared to the calibration standards. In each batch of tests, 0.173 ml of Perchloric acid and 26 g of ammonium sulfate are used.</p>	
<p><b>Frequency of Operation:</b> 1-4 times per year</p>	
<p><b>Environmental impact:</b></p> <p>At end of the test, the ammonium sulfate/perchlorate solutions are sink disposed with copious amounts of water for safety reasons. Disposal of the solutions by dilution in large amounts of water is by far the safest disposal method and has negligible environmental consequences. Once diluted into BNL's waste water system's volume, all risk of the reactivity/explosion hazard is eliminated. As per "Liquid Effluent Evaluation Form" dated 08/30/04, if analysis of field samples are conducted in a period when the BNL Sanitary treatment Plant is experiencing elevated levels of Perchlorates, permission for sink release will be suspended and the samples will need to be containerized for waste disposal. For PPE used during testing or analysis, washing the outer surfaces over a sanitary sink is appropriate. The PPE can then be disposed of in the non-hazardous trash.</p>	
<p><b>Waste Disposal:</b></p> <ul style="list-style-type: none"> <li>• Ammonium sulfate/perchlorate solutions are sink disposed with copious amounts of water for safety reasons. As per "Liquid Effluent Evaluation Form" dated 08/30/04, if analysis of field samples are conducted in a period when the BNL Sanitary treatment Plant is experiencing elevated levels of Perchlorates, permission for sink release will be suspended and the samples will need to be containerized for waste disposal.</li> <li>• PPE used during testing or analysis, washing the outer surfaces over a sanitary sink is appropriate. The PPE can then be disposed of in the non-hazardous trash.</li> </ul>	