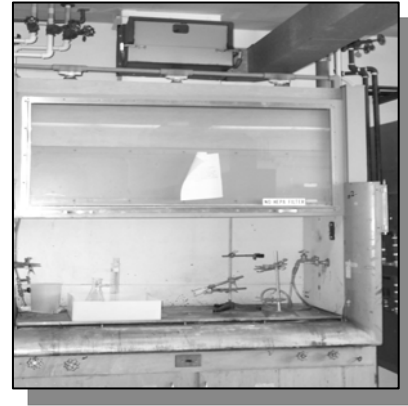


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## Content

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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 Group Leader who may delegate authority to administer this program. Members of the SHSD Industrial Hygiene Group can be qualified to perform certain tasks in this program. Personnel

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who have demonstrated competency in performing a certain role, in accordance with Section 7 of this procedure, will be qualified to serve that role.

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 using the SHSD IH 51300 *Chain of Custody* procedure. 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, in accordance with Section 7, to perform this field procedure.

**Qualified Analyzer:** A person who has demonstrated competency, in accordance with Section 7, to perform this analysis procedure.

### 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).

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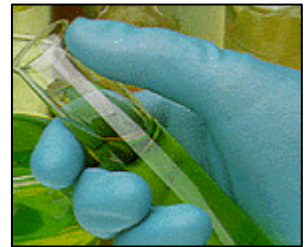
#### 4.2 Area Access:

- 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:

- 5.1.1 **During Sampling:** Appropriate personal protective equipment to protect the person collecting the sample must be used when implementing this procedure.
  - 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: At a minimum, 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. Disposable garments must be discarded as hazardous waste if contact with contamination has occurred. If personal clothing items become contaminated, they must be surrendered for BNL cleaning or disposal.
  - Foot: If contact of the feet is anticipated with contaminated surfaces, 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. If chemical or radiological levels from contamination in the area exceed or are likely to exceed the OSHA, ACGIH, or DOE standards, respirators are required. A half face or full face APR or PAPR respirator with appropriate cartridge or an air line respirator may be



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used up to the assigned protection factor listed in the BNL's Respiratory Protection Selection and Issuance SOPs.

- Eye: Safety Glasses with side shields are required in laboratories, construction, and general industry areas. When hazardous chemicals can significantly injure the eyes, a full face respirator must be used.

**5.1.2 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.

**5.2 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.

**5.3 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.



**Emergency:**

For small spills of perchloric acid/ perchlorate solution, squeegee liquid into a pan and sink dispose with copious amounts of water; use wet paper towels to clean all affected surfaces.

**5.4 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.

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**5.5 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 SHSD Operation Environmental Evaluation [SHSD-EMS-04](#):

“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. Policy is documented in IHG SOP IH75200. 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.”

**Waste Disposal:**

- 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.

**5.6 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

- PPE listed in Section 5.1.
- 4x4 inch cotton gauze or equivalent
- Rod or extension device
- Distilled water



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- Sample containers
- Rubber bands (to hold gauze or pad to extension device)

### Analysis Equipment

- PPE listed in Section 5.
- Ion selective electrode testing kit
- Water sprayer
- Suction pump or equivalent equipment
- Tygon tubing
- Flasks
- 2M Ammonium Sulfate solution (See 6.6 for preparation)
- Ion Buffer (See 6.7 for preparation)
- 2000 ppm Perchlorate Stock (See 6.10 for preparation)
- Orion Model 290A pH/mV meter
- Orion dual probes:
  - Yellow *Reference* probe
  - Black *Perchlorate* probe

## 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

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- 6.2.1 Secure a piece of gauze to a wooden rod or other appropriate extension device. A rubber band typically secures the gauzes well.
- 6.2.2 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.
- 6.2.3 Using gloves, remove the gauze and place into a labeled sample container.
- 6.2.4 Add **25mL** of distilled water.
- 6.2.5 Shake the vial for at least one minute.
- 6.2.6 Continue using Step 6.3.1 to 6.3.5 until all samples are collected.
- 6.2.7 Proceed to section 6.6 for analysis procedure.

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

- 6.3.1 Spray about 1 liter of distilled water into the fan housing or exhaust opening (system must be off).
- 6.3.2 Direct the spray toward the fan while someone slowly turns the fan shaft.
- 6.3.3 Allow the water to accumulate at the bottom of the fan housing (or in hood as appropriate).
- 6.3.4 Feed Tygon® tubing into the fan housing until the tube reaches the wash water.
- 6.3.5 Turn on a suction pump to collect the wash down water. It will provide a sample liquid (sufficient sample volume is 50mL).
- 6.3.6 Proceed to section 6.6 for analysis procedure.

- 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; 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

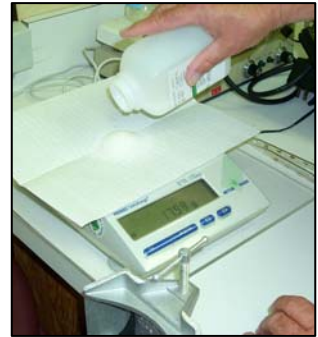
## Sample Analysis for Perchlorates

This method is applicable to aqueous solutions containing perchloric acid and its salts in the range of about 1 to 10,000 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 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.

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### 6.5 Ammonium Sulfate solution preparation

- 6.5.1 Tare a piece of weighing paper on the balance.
- 6.5.2 Weight **26.43** +/- 0.5 grams of Ammonium Sulfate on the balance.
- 6.5.3 Dissolve the 26.43g of Ammonium Sulfate (99+% purity) in approximately 50mL distilled water in a 100 ml beaker.
- 6.5.4 Transfer the solution to a 100mL volumetric flask.
- 6.5.5 Repeatedly rinse the transfer beaker with distilled water; transfer the rinses to the volumetric flask.
- 6.5.6 Bring flask up to the **100mL** mark with de-ionized or distilled water.
- 6.5.7 Stopper the flask and invert several times to mix.
- 6.5.8 Label flask as "**2M Ammonium Sulfate**" with the current date.



### 6.6 Ion Buffer solution preparation

- 6.6.1 Pipette 10mL of the *2M Ammonium Sulfate Solution* into a 100mL volumetric flask and fill to the mark with distilled water.
- 6.6.2 Label the flask "**0.2M Ion Buffer solution**". This solution will be used to fill the outer chamber of the Reference Electrode described in section 6.8 below.

### 6.7 Instrument set-up

- 6.7.1 **Battery Check:** On the Orion Model 290A pH/mV meter, check the battery status by turning the meter "on" (press the "**Power**" button). Replace the 9V-battery if no display occurs.
- 6.7.2 **Reference Electrode:** Flush the yellow, double junction reference electrode with distilled water. (Note: Refill each chamber with the appropriate solution before taking measurements.)
  - Fill the inner chamber with the manufacturer supplied "**Double Junction Reference Electrode Inner Filling Solution**", Orion Part Number 900002.
  - Fill the outer portion with the **Ion Buffer Solution** prepared in Step 6.7.2.
- 6.7.3 **Specific Ion (Perchlorate) Electrode:** Remove the rubber end-cap from the black *93 Series Electrode* body (if new or dry stored) or remove the probe from its long-term storage distilled water bath.



### 6.8 Establishing a Baseline Meter Reading:

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- 6.8.1 Place both probes into a 100 ml beaker containing the ion buffer solution to 1” to 2” depth. Insert a stirring bar and place the beaker and probes on a stirring plate. Spin the bar.
- 6.8.2 Turn the meter “on”. Use the “**mode**” button to select the “**mV**” setting. **If the reading does not stabilize and is NOT greater than 280mV, the Perchlorate probe is depleted and should be replaced.** Do not proceed with measurements using that probe.
- 6.8.3 Record the meter reading of the perchlorates in the *Ion Buffer (Pre-Test)* line on the *Perchlorate Analysis Record*.
- 6.8.4 Rinse the probes with distilled water between solutions.



#### 6.9 Perchlorate Stock solution preparation

- 6.9.1 Pipette **0.43mL** of 70% Perchloric acid into a 100mL volumetric flask.
- 6.9.2 Add distilled water to the **100mL** mark, cap the flask, and invert to mix.
- 6.9.3 Label as “*5000ppm Perchlorate Stock*” and mark the date on the flask.

#### 6.10 Calibration Standards preparation

- 6.10.1 Calibration Solution 1: Using a volumetric pipette, pipette **0.1mL** of the *5000 ppm-perchlorate stock* into a 50mL volumetric flask. Add **5mL** of 2M Ammonium Sulfate and fill to the mark with distilled water. Label as “*10 ppm-perchlorate calibration standard.*”
- 6.10.2 Calibration Solution 2: Using a volumetric pipette, pipette **1.0mL** of the *5000 ppm-perchlorate stock* into a 50mL volumetric flask. Add **5mL** of 2M Ammonium Sulfate and fill to the mark with distilled water. Label as “*100 ppm-perchlorate calibration standard.*”
- 6.10.3 Calibration Solution 3: Using a volumetric pipette, pipette **4.0mL** of the *5000 ppm-perchlorate stock* into a 50mL volumetric flask. Add **5mL** of 2M Ammonium Sulfate and fill to the mark with distilled water. Label as “*400 ppm-perchlorate calibration standard.*”
- 6.10.4 Calibration Solution 4: Add **10.0 mL** of the *5000 ppm-perchlorate stock* into a 50mL volumetric flask. Add 5 mL of 2M Ammonium Sulfate into a 50mL volumetric flask. and fill to the mark with distilled water. Label as “*1000 ppm-perchlorate calibration standard.*”

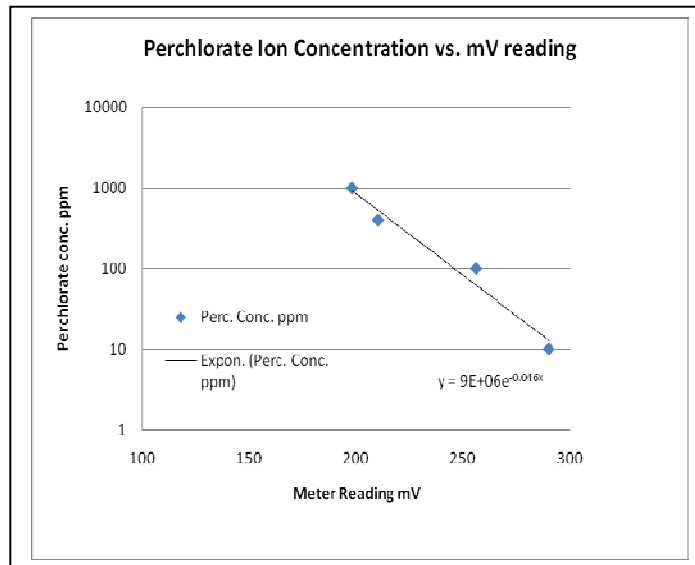
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| Final Concentration [ppm] | Volume of "5000 ppm Stock" | Volume of Distilled Water | Volume of 2M Ammonium Sulfate | Total Volume |
|---------------------------|----------------------------|---------------------------|-------------------------------|--------------|
| <b>0</b>                  | 0                          | 45.0 ml                   | 5.0 ml                        | 50 ml        |
| <b>10</b>                 | 0.1 ml                     | 44.9 ml                   | 5.0 ml                        | 50 ml        |
| <b>100</b>                | 1.0 ml                     | 44.0 ml                   | 5.0 ml                        | 50 ml        |
| <b>400</b>                | 4.0 ml                     | 41.0 ml                   | 5.0 ml                        | 50 ml        |
| <b>1000</b>               | 10.0 ml                    | 35.0 ml                   | 5.0 ml                        | 50 ml        |
| <b>4500</b>               | 45.0 ml                    | 0                         | 5.0 ml                        | 50 ml        |

#### 6.11 Calibration Curve analysis

- 6.11.1 Prepare a blank ("zero") sample using deionized (DI) water, which consists of 45mL DI water, 5mL 2M Ammonium Sulfate solution.
- 6.11.2 Pour each 50 ml calibration stock (or blank) solution into a separate, clean 100 ml beaker for analysis. Analyze the blank solution first and at the end of each sample set.
- 6.11.3 Starting with the lowest standard, immerse both electrodes into the beaker containing the calibration solution. Place a magnetic stirring bar in the solution. Stir the solution and allow the electrode reading to stabilize. Record the mV value on the *Perchlorate Analysis Record*.
- 6.11.4 Plot the points on a graph of "mV" versus "ppm concentration" in a log-Normal format. This will give a linear correlation for the analyses of samples with unknown concentrations.
- 6.11.5 Repeat calibration after every 10-12 field samples.

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Example calibration curve

### 6.12 Sample Analysis

- 6.12.1 Pipette **5mL** of sample solution from Step 6.3 into a 50mL volumetric flask.
- 6.12.2 Add **5mL** of **2M Ammonium Sulfate** solution.
- 6.12.3 Fill to the 50mL volume mark with distilled water and shake or swirl to mix.
- 6.12.4 Transfer to a 100mL beaker. Add a magnetic stir bar and immerse both electrodes into the solution. Spin the bar.
- 6.12.5 Allow the electrode reading to stabilize. Record the mV reading on the *Perchlorate Analysis Record*.
- 6.12.6 Rinse the electrodes with distilled water between samples.
- 6.12.7 Place the meter into the Ion Buffer solution. Record the meter reading of the perchlorates in the *Ion Buffer (Post-Test)* line on the *Perchlorate Analysis Record*.
- 6.12.8 Rinse the probes with distilled water.
- 6.12.9 Place the probes into distilled water and record the mV reading in the *Distilled Water Post-Test* line on the *Perchlorate Analysis Record*.
- 6.12.10 **With the mV reading and using the calibration curve obtained in Section 6.12 determine the concentration in ppm of each sample.**

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

- 6.13.1 **Samples with a concentration of less than 100 ppm in the test solution are considered negative for the risk of a fire or explosion hazard, i.e. no**

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perchlorates. Samples with a concentration of 100-500 ppm 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 greater than 500 ppm are considered positive for the presence of perchlorates.

- 6.13.2 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.

#### 6.14 Clean-up of lab equipment

- 6.14.1 Dilute reagents and field sample solutions with tap water and discharge to sink. Flush the sink with water for 3 minutes.
- 6.14.2 Wash glassware with detergent. Rinse glassware thoroughly with tap water. Triple rinse with distilled water. Air Dry.
- 6.14.3 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. Flush the sink with water for 3 minutes.
- 6.14.4 Rinse the outsides of the electrodes in distilled water. Store the electrodes in distilled water, covered in a manner to prevent evaporation of the water.**

### 7.0 Implementation and Training

- 7.1 **Qualification Criteria:** Only persons who have demonstrated competency in performing this test, to the satisfaction of the SHSD IH Group Leader or designee will be qualified to perform this test. The qualification criteria to perform this procedure are:
- 7.1.1 Knowledge of industrial hygiene practice (awareness level).
  - 7.1.2 Specific knowledge of this procedure.
  - 7.1.3 Demonstrated competency in performing this test via:
    - Visual observation of the sample wiping technique and/or analysis procedure.
    - Ability to answer questions on the sampling and analysis procedure and chain of custody of the sample during sampling and transportation.

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| SUBJECT:<br><br><b>Perchlorate and Perchloric Acid<br/>         Sampling and Analysis Procedure</b>   | DATE<br><b>07/21/09</b>       |
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- Knowledge of the appropriate personal protective equipment for the hazards of this particular type of sampling.

7.2 **Qualification Frequency & Record-keeping:** The SHSD IH Group Leader, or designee, will qualify SHSD IHG personnel to use this procedure. Personnel shall be re-qualified at a frequency not to exceed three years, provided there is no break in the work assignment that utilizes this procedure.

7.3 Personnel are to document their training using the Attachment 9.3 with its *Job Performance Measure Completion Certificate* for this meter. Qualification on this JPM is required on a 3 year basis, providing the professional is [performing this test](#) frequently.

## 8.0 References

- 8.1 *Perchloric Acid Contaminated Hood Decontamination Procedure Manual, Oak Ridge National Laboratory, Oak Ridge, TN, 1993.*
- 8.2 **Orion Research Instruction Manual for the Perchlorate Ion Electrode, 1990.**

## 9.0 Attachments

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

## 10.0 Documentation

| Document Development   |   |   |
|--|---|---|
| PREPARED BY:<br><i>(Signature and date on file)</i><br>A. Sells & R. Selvey<br>Date 03/01/00 | REVIEWED BY:<br><i>(Signature and date on file)</i><br>R. Selvey<br>SHSD IH Group Date 09/27/01 | APPROVED BY:<br><i>(Signature and date on file)</i><br>R. Selvey<br>SHSD IH Group Leader Date<br>09/28/01 |
| ESH Coordinator/ Date:<br><br><i>none</i>  | Work Coordinator/ Date:<br><br><i>none</i>  | SHSD Manager / Date<br><br><i>none</i>  |

The only official copy is on-line at the SHSD IH Group website.  
 Before using a printed copy, verify that it is current by checking the document issue date on the website.

|   |                               |
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|  |   |  |
|--|---|--|
| QA Representative / Date:<br><i>none</i>   | Training Coordinator / Date:<br><i>none</i>   | Filing Code:<br><b>IH52</b>  |
| Facility Support Rep. / Date:<br><i>none</i>   | Environ. Compliance Rep. / Date:<br><i>none</i>   | Effective Date:<br><b>09/28/01</b>   |
| ISM Review - Hazard Categorization<br><input checked="" type="checkbox"/> High<br><input type="checkbox"/> Moderate<br><input type="checkbox"/> Low/Skill of the craft | Validation:<br><input checked="" type="checkbox"/> Formal Walkthrough<br><input type="checkbox"/> Desk Top Review<br><input type="checkbox"/> SME Review<br>Name / Date: R. Selvey 7/2009 | Implementation:<br>Training Completed: Tracked in BTMS<br>Procedure posted on Web:07/21/09<br>Hard Copy files updated: 07/21/09<br>Document Control:07/21/09 |

| Revision Log  |
|---|
| Purpose: <input type="checkbox"/> Temporary Change <input type="checkbox"/> Change in Scope <input type="checkbox"/> Periodic review <input checked="" type="checkbox"/> Clarify/enhance procedural controls<br>Changed resulting from: <input type="checkbox"/> Environmental impacts <input type="checkbox"/> Federal, State and/or Local requirements <input type="checkbox"/> Corrective/preventive actions to non-conformances <input checked="" type="checkbox"/> none of the above<br>Section/page and Description of change: 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.<br>SME Reviewer/Date: (signature on file) 03/12/03 Robert Selvey |
| Purpose: <input type="checkbox"/> Temporary Change <input type="checkbox"/> Change in Scope <input type="checkbox"/> Periodic review <input checked="" type="checkbox"/> Clarify/enhance procedural controls<br>Changed resulting from: <input type="checkbox"/> Environmental impacts <input type="checkbox"/> Federal, State and/or Local requirements <input type="checkbox"/> Corrective/preventive actions to non-conformances <input checked="" type="checkbox"/> none of the above<br>Section/page and Description of change: Minor changes to Section 7. Minor format changes to Equipment section.<br>SME Reviewer/Date: (signature on file) R. Selvey 02/01/06  |
| Purpose: <input type="checkbox"/> Temporary Change <input type="checkbox"/> Change in Scope <input type="checkbox"/> Periodic review <input checked="" type="checkbox"/> Clarify/enhance procedural controls<br>Changed resulting from: <input type="checkbox"/> Environmental impacts <input type="checkbox"/> Federal, State and/or Local requirements <input type="checkbox"/> Corrective/preventive actions to non-conformances <input checked="" type="checkbox"/> none of the above<br>Section/page and Description of change: 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#.<br>SME Reviewer/Date: (signature on file) R. Selvey 05/23/07                                  |
| Purpose: <input type="checkbox"/> Temporary Change <input type="checkbox"/> Change in Scope <input type="checkbox"/> Periodic review <input checked="" type="checkbox"/> Clarify/enhance procedural controls<br>Changed resulting from: <input type="checkbox"/> Environmental impacts <input type="checkbox"/> Federal, State and/or Local requirements <input type="checkbox"/> Corrective/preventive actions to non-conformances <input checked="" type="checkbox"/> none of the above<br>Section/page and Description of change: Added Attachment 9.4. SME Reviewer/Date: (signature on file) R. Selvey 02/06/09  |
| Purpose: <input type="checkbox"/> Temporary Change <input type="checkbox"/> Change in Scope <input type="checkbox"/> Periodic review <input checked="" type="checkbox"/> Clarify/enhance procedural controls<br>Changed resulting from: <input type="checkbox"/> Environmental impacts <input type="checkbox"/> Federal, State and/or Local requirements <input type="checkbox"/> Corrective/preventive actions to non-conformances <input checked="" type="checkbox"/> none of the above<br>Section/page and Description of change: Updates 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.<br>SME Reviewer/Date: (signature on file) W. Litzke 07/21/09 and A. Meier 07/21/09       |

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|   | REVISION<br><b>Final Rev5</b> |
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## Attachment 9.1

### Sample of the Sampling & Analysis Record

|  |          |                         |  |                                 |   |
|--|----------|-------------------------|--|---------------------------------|---|
| <b>BROOKHAVEN NATIONAL LABORATORY</b><br>Safety and Health Services Division<br>INDUSTRIAL HYGIENE GROUP                               |          |                         | <b>IH75200</b><br><b>Attachment 9.1</b><br><i>Farm Revision 06/19/09</i> |                                 |   |
| <b>Perchlorate Sampling and Analysis Record</b>  |          |                         |  |                                 |   |
| <b>IH SERVICE NUMBER:</b> _____<br><b>Date:</b> _____<br><b>Samplers:</b> _____<br><b>Analysts:</b> _____<br><b>Building No:</b> _____ |          |                         | <b>STANDARDS</b>   | <b>Meter</b><br>Reading (mV)    | <b>Perchlorate</b><br>Concentration (ppm) |
| Perchlorate Concentration < 500 ppm  |          |                         | Blank/Zero (pre-test) 0 ppm  |                                 |   |
|  |          |                         | Standard 10 ppm  |                                 |   |
|  |          |                         | Standard 100 ppm   |                                 |   |
|  |          |                         | Standard 400 ppm   |                                 |   |
|  |          |                         | Standard 1000 ppm  |                                 |   |
| Room No:   | Hood No: | Location or Description | Meter Reading (mV)   | Perchlorate Concentration (ppm) |   |
| 1  |          |                         |  |                                 |   |
| 2  |          |                         |  |                                 |   |
| 3  |          |                         |  |                                 |   |
| 4  |          |                         |  |                                 |   |
| 5  |          |                         |  |                                 |   |
| 6  |          |                         |  |                                 |   |
| 7  |          |                         |  |                                 |   |
| 8  |          |                         |  |                                 |   |
| 9  |          |                         |  |                                 |   |
| 10   |          |                         |  |                                 |   |
| 11   |          |                         |  |                                 |   |
| 12   |          |                         |  |                                 |   |
|  |          |                         | Standard 10 ppm  | 204                             | 10  |
|  |          |                         | Standard 100 ppm   | 150                             | 100                                       |
|  |          |                         | Standard 400 ppm   | 115                             | 400                                       |
|  |          |                         | Standard 1000 ppm  | 90                              | 1000                                      |
| 13   |          |                         |  |                                 |   |
| 14   |          |                         |  |                                 |   |
| 15   |          |                         |  |                                 |   |
| 16   |          |                         |  |                                 |   |
| 17   |          |                         |  |                                 |   |
| 18   |          |                         |  |                                 |   |
| 19   |          |                         |  |                                 |   |
| 20   |          |                         |  |                                 |   |
| 21   |          |                         |  |                                 |   |
| 22   |          |                         |  |                                 |   |
| 23   |          |                         |  |                                 |   |
| 24   |          |                         |  |                                 |   |
|  |          |                         | Blank/Zero (post-test) 0 ppm   |                                 |   |
|  |          |                         | Standard 10 ppm  |                                 |   |
|  |          |                         | Standard 100 ppm   |                                 |   |
|  |          |                         | Standard 400 ppm   |                                 |   |
|  |          |                         | Standard 1000 ppm  |                                 |   |

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

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

### Practical Skill Evaluation: Field Sampling

| Criteria                                | Qualifying Performance Standard  | Unsatis-<br>factory | Reco-<br>vered | Satisf-<br>actory |
|---|--|---------------------|----------------|-------------------|
| <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. Personal Protective Equipment</b> | Understands the need to be aware of the potential surface contamination, airborne levels of contaminants, radiological hazards, and noise hazard. Knows how to determine the need for 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 self  |                     |                |                   |
|   | Knows the technique to lift the highest possible percentage of potential Perchlorates from the surface sampled   |                     |                |                   |
|   | Knows where to take sample to collect the highest possible sample concentration  |                     |                |                   |
| <b>5. Documentation</b>                 | Demonstrates correctly filling out IH forms, transfers appropriate info to IH databases, and prepares an evaluation assessment report.   |                     |                |                   |

### Lab Analysis Practical Skill Evaluation: Demonstration of Methodology

| Criteria   | Qualifying Performance Standard   | Unsatis-<br>factory | Reco-<br>vered | Satisf-<br>actory |
|--|---|---------------------|----------------|-------------------|
| <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. Personal Protective Equipment</b>          | Understands the need to be aware of the potential surface contamination and radiological hazards on submitted samples. Knows the need for 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 the AMS solution.  |                     |                |                   |
|  | Knows how to properly measure field samples with the conductivity meter.  |                     |                |                   |
|  | Knows how to properly determine the concentration of Perchlorates in the field 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> |
|-----------------------------|--------------|

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> |
|-----------------------------|--------------|

## 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>   |   |