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National Personal Protective Technology Laboratory
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Procedure No. CET-APRS-STP-CBRN-0309	Revision: 1.1	Date: 13 December 2005
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DETERMINATION OF CBRN HYDRIDE GASES (PHOSPHINE)
SERVICE-LIFE TEST, AIR-PURIFYING RESPIRATORS
STANDARD TEST PROCEDURE (STP)

1. PURPOSE

This test establishes the procedure for ensuring that the level of protection provided by the *CBRN Hydride Gases (Phosphine) Service Life, Air-Purifying Respirators Standard Test Procedure* submitted for Approval, Extension of Approval, or examined during Certified Product Audits meet the minimum certification standards set forth in *42 CFR, Part 84, Subpart G, Section 84.63(a)(c)(d)*; Volume 60, Number 110, June 8, 1995 and the *Statement of Standard for Chemical, Biological, Radiological, and Nuclear (CBRN) Full- Facepiece Air- Purifying Respirator (APR)* Dated March 7, 2003.

2. GENERAL

This STP describes the Determination of Phosphine Service Life Test, Air-Purifying Respirators test in sufficient detail that a person knowledgeable in the appropriate technical field can select equipment with the necessary resolution, conduct the test and determine whether the product passes the test.

3. EQUIPMENT AND MATERIAL

3.1. The list of necessary test equipment and materials follows:

3.1.1. Flow-Temperature-Humidity Control System or equivalent. Miller-Nelson (MN) control system, Model HCS-401. An automated system to control the airflow, temperature, and humidity of an air supply for an operating system. Laboratory air and distilled water are supplied to the unit; the three sensors and controlling mechanisms are incorporated electronically. The capacity of the unit is airflow: 20 to 200 liters / minute +/- 2%), relative humidity: 20 to 90 +/- 3 % and temperature ambient to 35 °C +/- 1°C.

Approvals:	<u>1st</u> Level	<u>2nd</u> Level	<u>3rd</u> Level
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- 3.1.2. EdgeTech Dew Prime II Hygrometer, Model 2000 or equivalent. A micro-processor based programmable chilled mirror dew point hygrometer. The hygrometer uses the dew point and ambient temperature to calculate the relative humidity. Ambient temperature range is: -50°C to $130^{\circ}\text{C} \pm 0.2^{\circ}\text{C}$; relative humidity is 1% to $95\% \pm 0.5\%$
- 3.1.3. Labview software developed for NIOSH Service Life Testing.
- 3.1.4. California Analytical Instruments, Model 1314 Photoacoustic Multigas Monitor with UA0970 optical filter or equivalent. This monitor uses photoacoustic infra-red detection to measure gases that absorb infra-red light. A filter is used to select the wavelength specific for the gas being detected. Range for phosphine is 10000 to 1.0 ppm ± 0.1 ppm.
- 3.1.5. CEA Model TG-4000BA Phosphine detector or equivalent. This detector uses electrochemical sensor for detection of phosphine. It has an internal vacuum pump for drawing the sample. Range is 0 to 1 ppm.
- 3.1.6. Mass Flow Controller, Brooks Instruments, variable flow rate depending on use.
- 3.1.7. Read out and Control Electronics, Brooks Instruments, Model 0154, Power supply and controller for the Brooks Mass Flow Controller or equivalent. The flow controllers are an integrally mounted control valve module with which stable gas flows can be achieved. Various flow rates are used with an accuracy of $\pm 0.7\%$ of rate and $\pm 0.2\%$ full scale.
- 3.1.8. Dry Gas Meter. Must have NIST traceable calibration certificate.
- 3.1.9. Certified cylinder of approximately 1 and 300 parts per million (ppm) PH_3 .
- 3.1.10. Phosphine cylinder, 99% purity, with regulator and purge valve.
- 3.1.11. Nitrogen cylinder, 99% purity.
- 3.2. Test fixture for mounting canisters.
- 3.3. The test chamber consisting of a 12" x 11½" x 7" air tight metal box with door opening lined with gasket material. Two ½" bulkhead Swagelok® fittings located on the backside of the test chamber for the introduction of the test concentration and for the exit of the test fixture. This fixture is not commercially available.
- 3.4. Resistance tester consisting of a vacuum source capable of delivering 85 Lpm, a

6-inch water column slant manometer or electronic manometer and connections appropriate for items being tested. See description in RCT-APR-003 and RCT-APR-007.

4. TESTING REQUIREMENTS AND CONDITIONS

- 4.1 This test procedure is only valid if the respirator system has first completed NIOSH Standard Test Procedure entitled Determination Of Durability Test For Environmental And Transportation Conditions And Rough Handling Drop Test On Chemical Biological Radiological Nuclear (CBRN) Air-Purifying Respirators (APR) And Canisters Standard Test Procedure (STP).
- 4.2 Prior to beginning any testing, all measuring equipment to be used must have been calibrated in accordance with the manufacturer's calibration procedure and schedule. At a minimum, all measuring equipment utilized for this testing must have been calibrated within the preceding 12 months using a method traceable to the National Institute of Standards and Technology (NIST).
- 4.3 Any laboratory using this procedure to supply certification test data to NIOSH will be subject to the provisions of the NIOSH Supplier Qualification Program (SQP). This program is based on the tenets of ISO/IEC 17025, the NIOSH Manual of Analytical Methods and other NIOSH guidelines. An initial complete quality system audit and follow on audits are requirements of the program. Additional details of the Program and its requirements can be obtained directly from the institute.
- 4.4 Precision and accuracy (P&A) must be determined for each instrument in accordance with laboratory procedures and NIOSH/NPPTL guidance. Sound practice requires, under *NIOSH Manual of Analytical Methods*, demonstrating a tolerance range of expected data performance of a plus or minus 25% of a 95% confidence interval of the stated standard requirement. NIOSH/NPPTL P&A tolerance can be higher but not lower.
- 4.5 Compressed gas cylinders must meet all applicable Department of Transportation requirements for cylinder approval as well as retesting / requalification.
- 4.6 Normal laboratory safety practices must be observed. This includes safety precautions described in the current NIOSH Pittsburgh Health and Safety Program.
 - 4.6.1 Safety glasses, lab coats and hard-toe shoes must be worn at all times.
 - 4.6.2 Workbenches must be maintained free of clutter and non-essential test equipment.

4.6.3 When handling any broken glass laboratory equipment, lab technicians and personnel must wear special gloves, which protect against lacerations or punctures.

4.6 Please refer to Material Safety Data Sheets and the NIOSH Health and Safety Manual for the proper protection and care in handling, storing, and disposing of the chemicals and gases used in this procedure.

5. PROCEDURE

Note: Reference Section 3 for equipment, model numbers and manufacturers. For calibration purposes use those described in the manufacturer's operation and maintenance manuals.

- 5.1. Follow individual instruction manuals for set up and maintenance of equipment used in this procedure prior to beginning testing. Malfunctioning equipment must be repaired or replaced and properly set up and calibrated before starting all tests.
- 5.2. Calibrate photoacoustic monitor for phosphine the challenge concentration of approximately 300 ppm.
- 5.3. Calibrate the CEA phosphine monitor for the breakthrough concentration of approximately 0.3 ppm.
- 5.4. Set up test equipment as shown in Figure 1. The humidity reading controlled by the Miller Nelson system and monitored the Dew Point Hygrometer. The sample pickup for the hygrometer is place into the air stream via a tee after the Miller Nelson and before the introduction point of challenge agent. The thermocouple for the hygrometer is placed in the challenge gas stream immediately before the test chamber.
- 5.5. Verify the following equipment is on:
 - 5.5.1. Miller Nelson Unit.
 - 5.5.2. Air and water supplies.
 - 5.5.3. NIOSH Service Life Apparatus Controller software program.
- 5.6. Establish the correct humidity and temperature as per the test standard in paragraph 6.3
- 5.7. Set the airflow to the required airflow for the test and the number of test being run at one time. Verify the airflow from the test fixture using the appropriate dry test

meter.

- 5.8. Weigh and record initial weight of the test canister on Test Data Sheet.
- 5.9. Take initial inhalation and exhalation resistances of the canister mounted on the facepiece and canister alone as described in RCT-APR-003 and RCT-APR-007. Record the values on Test Data Sheet.
- 5.10. Make sure the diverter valve in the system is diverting the challenge concentration airflow to discharge and not into the testing chamber.
- 5.11. Mount canister onto test fixture and place in testing chamber.
- 5.12. At the phosphine regulator, using the purge valve introduce dry nitrogen into the challenge introduction for five minutes. This should purge any contaminants and moisture from the phosphine introduction system. The system should be clear of residual moisture.
- 5.13. Start Nitrogen Dilution mass flow controller and set flow rate.
- 5.14. Miller Nelson Unit with the humidity turned down to zero. Set airflow to system.
- 5.15. Check the airflow from at the test fixture using the dry test meter.
- 5.16. Shut off nitrogen purge flow, open Phosphine cylinder with phosphine mass flow controller set at "0". Gradually, open mass flow controller to establish the test concentration for 300 ppm Phosphine in 64 or 100 liters per minute air.
- 5.17. Connect tubing from the tee in the airflow line to the phosphine challenge detector.
- 5.18. Once the test concentration of 300 ppm has been established, testing may begin.
- 5.19. Direct challenge concentration airflow into test chamber.
- 5.20. Start timer. Monitor effluent concentration. Monitor upstream and downstream temperatures throughout testing, record beginning and final temperatures on the test data sheet.
- 5.21. Run test until breakthrough of 0.3 ppm is observed or minimum service life is surpassed.
- 5.22. Direct challenge concentration airflow out of the test canister.

- 5.23. Weigh and record final weight of the test canister on Test Data Sheet.
- 5.24. Take initial inhalation and exhalation resistances of the canister mounted on the facepiece and canister alone as described in RCT-APR-003 and RCT-APR-007. Record the values on Test Data Sheet.
- 5.25. Allow clean air to purge through the test chamber.
- 5.26. Repeat steps 5.6 through 5.25 for each test described in section.
- 5.27. When all tests are completed, turn off phosphine cylinder, set temperature and humidity to zero and allow air to pass through the system for 30 minutes.
- 5.28. Open dry nitrogen Purge Valve on regulator, to clear system of residual phosphine. Allow nitrogen to purge through system for 10 - 15 minutes.

6. PASS OR FAIL CRITERIA

- 6.1. The criterion for passing this test is set forth in 42 CFR, Part 84, Subpart G, Section 84.63(a)(c)(d) and Subpart I, Section 84.110(c); Volume 60, Number 110, June 8, 1995.
- 6.2. This test establishes the standard procedure for ensuring that:
 - 84.63 Test requirements; general.
 - (a) Each respirator and respirator component shall when tested by the applicant and by the Institute, meet the applicable requirements set forth in subparts H through L of this part.
 - (c) In addition to the minimum requirements set forth in subparts H through L of this part, the Institute reserves the right to require, as a further condition of approval, any additional requirements deemed necessary to establish the quality, effectiveness, and safety of any respirator used as protection against hazardous atmospheres.
 - (d) Where it is determined after receipt of an application that additional requirements will be required for approval, the Institute will notify the applicant in writing of these additional requirements, and necessary examinations, inspections, or tests, stating generally the reasons for such requirements, examinations, inspections, or tests.
- 6.3. Hydride Gases (Phosphine) test for CBRN canisters

- 6.3.1. Resistance to airflow of both canister and system will be taken before and after each test.
- 6.3.2. Three canisters will be tested at 64 Lpm, continuous airflow, 25 % \pm 5 % relative humidity (RH), 25 °C \pm 5 °C and 300 ppm phosphine. Minimum service life will be 15, 30, 45, 60, 90 or 120 minutes as per manufacturer request.
- 6.3.3. Three canisters will be tested at 64 Lpm, continuous airflow, 80 % \pm 5 % relative humidity (RH), 25 °C \pm 5 °C and 300 ppm phosphine. Minimum service life will be 15, 30, 45, 60, 90 or 120 minutes as per manufacturer request.
- 6.3.4. Three canisters will be tested at 100 liters / minute, continuous airflow, 50 % \pm 5 % relative humidity (RH), 25 °C \pm 5 °C and 300 ppm phosphine. Minimum service life must be 5 minutes.
- 6.3.5. End of service life concentration is 0.3 ppm phosphine.

7. RECORDS AND TEST SHEETS

- 7.1. All test data will be recorded on the PHOSPHINE SERVICE LIFE test data sheet.
- 7.2. All videotapes and photographs of the actual test being performed, or of the tested equipment shall be maintained in the task file as part of the permanent record.
- 7.3. All equipment failing any portion of this test will be handled as follows:
 - 7.3.1. If the failure occurs on a new certification application, or extension of approval application, send a test report to the RCT Leader and prepare the hardware for return to the manufacturer.
 - 7.3.2. If the failure occurs on hardware examined under an Off-the-Shelf Audit the hardware will be examined by a technician and the RCT Leader for cause. All equipment failing any portion of this test may be sent to the manufacturer for examination and then returned to NIOSH. However, the hardware tested shall be held at the testing laboratory until authorized for release by the RCT Leader, or his designee, following the standard operating procedures outlined in Procedure for Scheduling, and Processing Post-Certification Product Audits, RB-SOP-0005-00.

8. ATTACHMENTS

- 8.1 Bench Top Set-Up

8.2 Data Sheet.

Revision History

Revision	Date	Reason for Revision
00	20 September 2004	Historic document
1.1	13 December 2005	Update header and format No changes to method