



National Institute for Occupational Safety and Health  
National Personal Protective Technology Laboratory  
P.O. Box 18070  
Pittsburgh, PA 15236

|                                      |               |                        |
|--------------------------------------|---------------|------------------------|
| Procedure No. CET-APRS-STP-CBRN-0404 | Revision: 1.1 | Date: 21 December 2005 |
|--------------------------------------|---------------|------------------------|

DETERMINATION OF CBRN ACID GASES (PHOSGENE)  
SERVICE-LIFE TEST, AIR-PURIFYING ESCAPE RESPIRATORS  
STANDARD TEST PROCEDURE (STP)

1. PURPOSE

This test establishes the procedure for ensuring that the level of protection provided by the *CBRN Acid Gases (Phosgene) Service Life Test, Air Purifying Escape 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, 84.63(a)(c)(d); Volume 60, Number 110, June 8, 1995 and the *Statement of Standard for Chemical, Biological, Radiological, and Nuclear (CBRN) Air Purifying Escape Respirator*, Dated September 30, 2003.

2. GENERAL

This STP describes the Determination of Phosgene Service Life Test, Air-Purifying Escape 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 or not the product passes the test.

3. EQUIPMENT AND MATERIAL

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

3.1.1. Miller Nelson Research Model 401 Flow-Temperature-Humidity Control System or equivalent. This system is 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 unit output is air of the variable volume/flow dependant on the size of unit (10% of max flow to max flow in liters per minute (Lpm)  $\pm$  2%), and relative humidity (10%–98%  $\pm$  3%) and temperature (20°C–30°C  $\pm$  0.3%).

|            |                  |                  |                  |
|------------|------------------|------------------|------------------|
| Approvals: | <u>1st</u> Level | <u>2nd</u> Level | <u>3rd</u> Level |
|------------|------------------|------------------|------------------|

- 3.1.2 EdgeTech Dew Prime II Hygrometer, Model 2000 or equivalent. A micro-processor based programmable dew point instrument. The hygrometer uses the dew point and ambient temperature to calculate the relative humidity. Dew point and ambient temperature range is: -50 to 130 °C  $\pm$  0.2 °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 UA0979 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 phosgene is 2000 to 0.02 ppm  $\pm$  0.002 ppm.
- 3.1.5 Mass Flow Controller, Brooks Instruments, variable flow rate depending on use.
- 3.1.6 Read out and Control Electronics, Brooks Instruments, Model 0154, Power supply and controller for the Brooks Mass Flow Controller. 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.
- 3.1.7 Dry Gas Meter. Must have NIST traceable calibration certificate.
- 3.1.8 Certified cylinder of 1.25, 125 and 250 parts per million (ppm) phosgene in Nitrogen.
- 3.1.9 Phosgene cylinder, 1 – 5 % balance nitrogen.
- 3.1 Test fixture for mounting canisters.
- 3.2 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.
- 3.3 Resistance tester consisting of a vacuum source capable of delivering 85 liters per minute, a 6-inch water column slant manometer and a 29/42 female ground glass joint.

#### 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 Escape Respirators 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. Work benches must be maintained free of clutter and non-essential test equipment.
  - 4.6.3. When handling any glass laboratory equipment, lab technicians and personnel must wear special gloves, which protect against lacerations or punctures.
- 4.7. **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. After the manufacturer's specified warm-up period, calibrate both photoacoustic monitors using the certified phosgene calibration gas cylinders. One monitor is calibrated for the challenge concentration of approximately 125 ppm for the general category standard or at approximately 250 ppm for the specific category standard. A second monitor is calibrated for the breakthrough concentration of approximately 1.25 ppm.
- 5.3. 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 placed 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.
- 5.4. Verify the following equipment is on:
  - 5.4.1. Miller Nelson Unit.
  - 5.4.2. Air and water supplies.
  - 5.4.3. Photoacoustic multi-gas monitors.
  - 5.4.4. NIOSH Service Life Apparatus Controller software program.
- 5.5. Establish the correct humidity and temperature as per the test standard in paragraph 6.3.
- 5.6. Set the airflow to the required airflow for the test. Verify the airflow from the test fixture using the appropriate dry test meter.
- 5.7. Weigh and record initial weight of the test canister on Test Data Sheet.

- 5.8. Take initial inhalation and exhalation resistances of the canister mounted on the facepiece at 85 liters per minute. Record the values on Test Data Sheet.
- 5.9. Make sure diverter valve in the system is diverting the challenge concentration airflow to discharge and not into testing chamber.
- 5.10. Mount canister onto test fixture and place in testing chamber.
- 5.11. Ensure that the nitrogen gas is ready for immediate flushing of regulator through the purge valve.
- 5.12. Turn on phosgene cylinder.
- 5.13. Establish the test concentration of 125 ppm, for the general category standard, or 250 ppm, for the specific category standard, phosgene.
- 5.14. Once the phosgene concentration has been established, testing may begin.
- 5.13 Direct challenge concentration airflow into test chamber.
- 5.14 Start timer. Airflow out of the fixture is directed into the breakthrough detector. Monitor and record the upstream and downstream temperatures of the air stream throughout testing. Record breakthrough values and times.
- 5.15 Run test until breakthrough of 1.25 ppm is observed or minimum service life is surpassed. Record this data on the test data sheet.
- 5.16 Direct challenge concentration airflow out of the test chamber.
- 5.17 Weigh and record final weight of the test canister on Test Data Sheet. Turn off phosgene cylinder.
- 5.17 Weigh and record final weight of the test canister on Test Data Sheet.
- 5.18. Take final inhalation and exhalation resistances of the canister mounted on the facepiece at 85 liters per minute. Record the values on Test Data Sheet.
- 5.19. Allow clean air to purge through test chamber for 5 minutes.
- 5.20. Repeat steps 5.5 through 5.19 for each test described in section 6.3.
- 5.21. When testing is complete turn off phosgene cylinder, purge regulator with dry nitrogen for 10-15 minutes. Set temperature and humidity to zero and allow air to

pass through the system for 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), Subpart I, Section 84.126, Subpart L, Section 84.207, and Subpart KK, Section 84.1157; 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. PHOSGENE BENCH TEST FOR CANISTERS.

### 6.3.1. General Category Standard.

- 6.3.1.1. Resistance to airflow of both canister and system will be taken before and after each test.
- 6.3.1.2. Three canisters will be tested at 64 Lpm, continuous air flow, 25 %  $\pm$  5 % relative humidity (RH), 25 ° C  $\pm$  5 ° C, and 125 ppm phosgene. Minimum service life will be 15, 30, 45, 60 or 90 minutes as per manufacture request.
- 6.3.1.3. Three canisters will be tested at 64 Lpm, continuous air flow, 80 %  $\pm$  5 % relative humidity (RH), 25 ° C  $\pm$  5 ° C, and 125 ppm phosgene. Minimum service life will be 15, 30, 45, 60 or 90 minutes as per manufacture request.

6.3.1.4. Three canisters will be tested at 100 Lpm, continuous air flow, 50 %  $\pm$  5 % relative humidity (RH), 25 ° C  $\pm$  5 ° C, and 125 ppm phosgene. Minimum service life of 5 minutes.

6.3.1.5. End of service life concentration is 1.25 ppm phosgene.

6.3.2. Specific Category Standard.

6.3.2.1. Resistance to airflow of both canister and system will be taken before and after each test.

6.3.2.2. Three canisters will be tested at 64 Lpm, continuous air flow, 25 %  $\pm$  5 % relative humidity (RH), 25 ° C  $\pm$  5 ° C, and 250 ppm phosgene. Minimum service life will be 15, 30, 45, 60 or 90 minutes as per manufacture request.

6.3.2.3. Three canisters will be tested at 64 Lpm, continuous air flow, 80 %  $\pm$  5 % relative humidity (RH), 25 ° C  $\pm$  5 ° C, and 250 ppm phosgene. Minimum service life will be 15, 30, 45, 60 or 90 minutes as per manufacture request.

6.3.2.4. Three canisters will be tested at 100 Lpm, continuous air flow, 50 %  $\pm$  5 % relative humidity (RH), 25 ° C  $\pm$  5 ° C, and 250 ppm phosgene. Minimum service life of 5 minutes.

6.3.2.5. End of service life concentration is 1.25 ppm phosgene.

7. RECORDS/TEST SHEETS

7.1. All test data will be recorded on the PHOSGENE 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 CET Team 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 CET Team 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 CET Team 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

| <b>Revision</b> | <b>Date</b>       | <b>Reason for Revision</b>                       |
|-----------------|-------------------|--|
| 0               | 21 September 2004 | Historic document                                |
| 1.1             | 21 December 2005  | Update header and format<br>No changes to method |
|                 |                   |  |
|                 |                   |  |