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National Personal Protective Technology Laboratory
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Procedure No. CET-APRS-STP-CBRN-0403	Revision: 1.1	Date: 21 December 2005
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DETERMINATION OF CBRN ACID GASES (HYDROGEN CYANIDE)
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 (Hydrogen cyanide) 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 CBRN Acid Gases (Hydrogen Cyanide) Service Life Test, Air-Purifying Escape Respirators Standard Test Procedure* 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/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
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- 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 UA0980 optical filter or equivalent.
 - 3.1.5. Gas Chromatograph, Shimadzu, Model 14A with Flame Ionization Detectors or equivalent.
 - 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.
 - 3.1.8. Dry Test Meter, American Meter Company, Model and size depending on air flow to be measured.
 - 3.1.9. Certified cylinders of 10 ppm, 500 ppm and 1000 ppm hydrogen cyanide, balance gas Nitrogen.
 - 3.1.10. Hydrogen cyanide permeation tube.
 - 3.1.11. Cyanogen permeation tube.
 - 3.1.12. Hydrogen Cyanide cylinders, 2% hydrogen cyanide, balance gas of helium.
- 3.1 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® fitting 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 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 broken 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. Calibrate photoacoustic monitor for hydrogen cyanide using a certified hydrogen cyanide gas cylinder. For the general category standard the monitor is calibrated for the challenge concentration of 470 ppm. For the specific category standard the monitor is calibrated for the challenge concentration of 940 ppm.
- 5.3 After the manufacturer's specified warm-up period, calibrate the Gas Chromatograph using the Kin-Tek gas generator with the hydrogen cyanogen and cyanogen permeation tube.
- 5.4 Start automatic sampling mode on the gas chromatograph. Monitor and record the upstream and downstream concentrations.
- 5.5 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.
- 5.6 Verify the following equipment is on:
- 5.6.1 Miller Nelson Unit.
 - 5.6.2 Air and water supplies.
 - 5.6.3 Gas Chromatograph
 - 5.6.4 NIOSH Service Life Apparatus Controller software program.

- 5.7 Establish the correct humidity and temperature using chilled mirror dew point hygrometer per the test standard in paragraph 6.3.
- 5.8 Set the airflow to the required airflow for the test. Verify the airflow from the test fixture using the appropriate dry test meter.
- 5.9 Weigh and record initial weight of the test canister on Test Data Sheet.
- 5.10 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.
- 5.11 Make sure the diverter valve in the system is diverting the challenge concentration airflow to the discharge and not into the test chamber Turn on the hydrogen cyanide cylinder.
- 5.12 Mount canister onto test fixture in testing chamber.
- 5.13 Turn on the hydrogen cyanide cylinder.
- 5.14 Establish the test concentration of 470 ppm hydrogen cyanide for general category standard or 940 ppm hydrogen cyanide for specific category standard.
- 5.15 Once the hydrogen cyanide concentration has been established, testing may begin.
- 5.16 Direct challenge concentration airflow into test chamber
- 5.17 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.
- 5.18 Run test until breakthrough of 10 ppm hydrogen cyanide and cyanogen is observed or minimum service life is surpassed.
- 5.19 Direct challenge concentration airflow out of the test chamber.
- 5.20 Weigh and record final weight of the test canister on Test Data Sheet.
- 5.21 Allow clean air to purge through the test chamber for 5 minutes.
- 5.22 Repeat steps 5.7 through 5.22 for each test described in section 6.3.
- 5.23 End sampling mode program in the gas chromatograph.

5.24 When all tests are complete turn off hydrogen cyanide cylinder, clean regulator by purging with air, set temperature and humidity to zero and allow air to pass through the system for 30 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 HYDROGEN CYANIDE 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 airflow, 25 % \pm 5 % relative humidity (RH), 25 °C \pm 5 °C, containing 470 ppm hydrogen cyanide Minimum service life will be 15, 30, 45 or 60 minutes as per manufacturer request.

6.3.1.3. Three canisters will be tested at 64 Lpm, continuous airflow, 80 % \pm 5 % relative humidity (RH), 25 °C \pm 5 °C, containing 470

ppm hydrogen cyanide. Minimum service life will be 15, 30, 45 or 60 minutes as per manufacturer request.

6.3.1.4. Three canisters will be tested at 100 Lpm, continuous airflow, 50 % \pm 5 % relative humidity (RH), 25 °C \pm 5°C, containing 470 ppm hydrogen cyanide. Minimum service life for all canisters will be 5 minutes.

6.3.1.5. End of service life concentration is 10 ppm, which is the sum of the breakthrough products of hydrogen cyanide and cyanogen.

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 airflow, 25 % \pm 5 % relative humidity (RH), 25 °C \pm 5 °C, containing 940 ppm hydrogen cyanide Minimum service life will be 15, 30, 45 or 60 minutes as per manufacturer request.

6.3.2.3. Three canisters will be tested at 64 Lpm, continuous airflow, 80 % \pm 5 % relative humidity (RH), 25 °C \pm 5°C, containing 940 ppm hydrogen cyanide. Minimum service life will be 15, 30, 45 or 60 minutes as per manufacturre request.

6.3.2.4. Three canisters will be tested at 100 Lpm, continuous airflow, 50 % \pm 5 % relative humidity (RH), 25 °C \pm 5°C, containing 940 ppm hydrogen cyanide. Minimum service life for all canisters will be 5 minutes.

6.3.2.5. End of service life concentration is 10 ppm, which is the sum of the breakthrough products of hydrogen cyanide and cyanogen.

7. RECORDS AND TEST SHEETS

7.1. All test data will be recorded on the CBRN HYDROGEN CYANIDE 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 Section Chief 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 Section Chief 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 Section Chief, 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	21 September 2004	Historic document
1.1	21 December 2005	Update header and format No changes to method