

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-0308

Revision: 1.1 Date: 13 December 2005

DETERMINATION OF CBRN NITROGEN OXIDE GASES (NITROGEN DIOXIDE) SERVICE-LIFE TEST, AIR-PURIFYING RESPIRATORS STANDARD TEST PROCEDURE (STP)

1. <u>PURPOSE</u>

This test establishes the standard procedure for ensuring that the level of protection provided by *CBRN Nitrogen Oxide Gases* (*Nitrogen Dioxide*) Service Life Test, Air-Purifying Respirators Standard Test Procedure submitted for Approval, Extension of Approval, or examined during Certified Product Audits meet the certification requirements 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 CBRN Nitrogen Dioxide Service Life Test, Air-Purifying Respirators Standard Test Procedure* 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) ± 2%), and relative humidity (10%–98% ± 3%) and temperature (20°C–30° °C ± 0.3%).

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

- 3.1.2 EdgeTech Dew Prime II Hygrometer, Model 2000 or equivalent. A microprocessor 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°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 Interscan Corporation Model RM-15-0 nitrogen dioxide detector or equivalent. This detector is an electrochemical voltammetric sensor. Detector range: 0 1999 ppm, resolution: 1 ppm.
- 3.1.5 Interscan Corporation Model RM-15-2 nitrogen dioxide detector or equivalent. This detector is an electrochemical voltammetric sensor. Detector range: 0 19.99 ppm, resolution: 0.01 ppm.
- 3.1.6 Interscan Corporation Model RM-54-1 nitrogen oxide detector or equivalent. This detector is an electrochemical voltammetric sensor. Detector range: 0 199.9 ppm, resolution: 0.1 ppm.
- 3.1.7 Mass Flow Controller, Brooks Instruments, variable flow rate depending on use.
- 3.1.8 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.
- 3.1.9 Dry Gas Meter. Must have NIST traceable calibration certificate.
- 3.1.10 Certified cylinders of 200 parts per million and 1 parts per million nitrogen dioxide in nitrogen.
- 3.1.11 Certified cylinders of 25 parts per million nitrogen oxide in nitrogen.
- 3.1.12 Nitrogen dioxide cylinder, 2.5 percent 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 Swaglok® 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.
- 4.7 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, adjust instruments to read zero and make adjustment for Labview software to read 0 ppm. Calibrate both nitrogen dioxide analyzers and the nitrogen oxide analyzer using the certified gas cylinder containing the 1 ppm or 200 ppm nitrogen dioxide and 25 ppm nitrogen oxide. During calibration make adjustment for Labview software to read the appropriate concentration for each detector.
- 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 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.
- 5.4 Verify the following equipment is on:
 - 5.4.1 Miller Nelson Unit.
 - 5.4.2 Air and water supplies.
 - 5.4.3 NIOSH Service Life Apparatus Controller software program.
 - 5.4.4 Nitrogen dioxide cylinder, 2.5 percent purity.

- 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 and the number of test being run at one time. 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.
- 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.
- 5.9 Mount canister onto test fixture and place in testing chamber.
- 5.10 Divert 0.5 Lpm airflow from airflow line to the nitrogen dioxide challenge detector.
- 5.11 Ensure that the nitrogen gas is ready for immediate flushing of regulator through the purge valve.
- 5.12 Turn on nitrogen dioxide cylinder.
- 5.13 Establish the test concentration of 200 ppm nitrogen dioxide.
- 5.14 Direct challenge concentration airflow into test chamber.
- 5.15 Start timer. Monitor effluent concentration using both the nitrogen dioxide and nitrogen oxide analyzers. Monitor upstream and downstream temperatures throughout testing, record beginning and final temperatures on the test data sheet.
- 5.16 Run test until breakthrough of 1 ppm nitrogen dioxide or 25 ppm nitrogen oxide is observed or minimum service life is surpassed. Record this data on the test data sheet.
- 5.17 Direct challenge concentration airflow away from test chamber.
- 5.18 Weigh and record final weight of the test canister on Test Data Sheet.
- 5.19 Take final 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.

- 5.20 Repeat steps 5.7 through 5.21 for each test described in section 6.3.
- 5.21 Allow clean air to purge through system for 10 15 minutes.

6. PASS/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); 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 Nitrogen oxide gases (Nitrogen dioxide) 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 air flow, 25 % \pm 5 % relative humidity (RH), 25 °C \pm 5 °C, and 200 ppm nitrogen dioxide. 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 air flow, $80\% \pm 5\%$ relative humidity (RH), 25 °C ± 5 °C, and 200 ppm nitrogen dioxide. 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 Lpm, continuous air flow, 50 % ± 5 % relative humidity (RH), 25 °C ± 5 °C, and 200 ppm nitrogen dioxide. Minimum service life of 5 minutes.
- 6.3.5 End of service life concentration is 1 ppm nitrogen dioxide or 25 ppm nitric oxide.

7. RECORDS AND TEST SHEETS

- 7.1. All test data will be recorded on the NITROGEN DIOXIDE 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.

Procedure No. CET-APRS-STP-CBRN-0308	Revision: 1.1	Date: 13 December 2005	Page 8 of 8
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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