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Procedure No. CET-APRS-STP-CBRN-0301	Revision: 1.1	Date: 29 June 2005
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DETERMINATION OF CBRN ORGANIC VAPOR (CYCLOHEXANE)  
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 Organic Vapor (Cyclohexane) 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 Organic Vapor (Cyclohexane) 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 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
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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:  $-50^{\circ}\text{C}$  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 Miran Ambient Air Analyzer, Model 1A with closed loop system or equivalent. The analyzer is a single beam variable filter spectrometer with a variable pathlength gas cell. For cyclohexane detection, the wavelength is set to  $3.4\ \mu\text{m}$  and the pathlength to 20.25 meters. Range is 300 to 0.03 ppm. The closed loop system consists of a stainless steel bellows pump, septum and tubing with a sample volume of 5.64 liters.
- 3.1.5 Amersham Biosciences High Precision Pump, Model P-500 with reservoir. Flow rate range: 1 ml/hr to 400 ml/hr  $\pm 1.5\%$  of setting.
- 3.1.6 Electronic Balance with an accuracy of 0.01 grams.
- 3.1.7 Mass Flow Controller, Brooks Instruments, model 5851. 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 Test Meter, American Meter Company, Model and size depending on air flow to be measured.
- 3.1.10 Miller Nelson Research, Model SV-2000, Vaporizer system or equivalent. Used for vaporizing cyclohexane solution.
- 3.1.11 Cyclohexane, ACS grade, 99%.
- 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, Transportation And Rough Handling Conditions On Chemical Biological Radiological Nuclear (CBRN) Full-Facepiece Air-Purifying Respirators (APR) 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 Adjust instrument to read zero and make adjustment for Labview software to read 0.0 ppm.

5.3. Verify Infrared Analyzer: Refer to the performance verification procedures in the user manual. Inject 0.25  $\mu$ L of cyclohexane into closed loop system of the IR as described in the manual. Once stabilized, the analyzer should read 10 ppm. Make adjustment for Labview software to read 10.0 ppm. Disconnect closed loop system and allow IR to return to zero reading.

5.4 Set the high precision pump for delivery of calculated cyclohexane to obtain 2600 ppm at the testing airflow. See appendix 8.2.

5.5 Set up test equipment as shown in Figure 1. The humidity reading is controlled by the Miller Nelson system and monitored by 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.

5.6. Verify the following equipment is on:

5.6.1 Miller Nelson Flow-Temperature-Humidity Control System.

- 5.6.2 Air and water supplies.
- 5.6.3 High precision pump.
- 5.6.4 Electronic balance.
- 5.6.5 NIOSH Service Life Apparatus Controller software program.
- 5.7 Establish the correct humidity and temperature as 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 Fill pump reservoir with cyclohexane. Insert needle from pump to septum tee in airline.
- 5.10 Weigh and record initial weight of the test canister on Test Data Sheet.
- 5.11 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.
- 5.12 Make sure diverter valve in the system is diverting the challenge concentration airflow to discharge and not into the testing chamber.
- 5.13 Mount canister onto test fixture in testing chamber.
- 5.14 Start flow from the high precision pump to the needle
- 5.15 Record the initial weight of the cyclohexane reserve.
- 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.
- 5.18 Run test until breakthrough of 10 ppm is observed or requested minimum service life is surpassed. Weigh and record the final weight of the cyclohexane reservoir.
- 5.19 Direct challenge concentration airflow out of test chamber.
- 5.20 Calculate and record the challenge concentration of the cyclohexane, see

attachment 8.2.

- 5.21 Weigh and record final weight of the test canister on Test Data Sheet.
- 5.22 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.
- 5.23 Allow clean air to purge through test chamber for 5 minutes.
- 5.24 Repeat steps 5.7 through 5.21 for each test described in section 6.3.
- 5.25 When all tests are completed turn off cyclohexane flow, 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)*; 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. Organic Vapor (Cyclohexane) 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),  $25^{\circ}\text{C} \pm 5^{\circ}\text{C}$  and 2600 ppm cyclohexane. 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,  $80\% \pm 5\%$  RH,  $25^{\circ}\text{C} \pm 5^{\circ}\text{C}$  and 2600 ppm cyclohexane. 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,  $50\% \pm 5\%$  RH,  $25^{\circ}\text{C} \pm 5^{\circ}\text{C}$  and 2600 ppm cyclohexane. Minimum service life must be 5 minutes.
- 6.3.5. End of service life concentration is 10 ppm cyclohexane.

## 7. RECORD AND TEST SHEETS

- 7.1. All test data will be recorded on the CBRN Organic Vapor (Cyclohexane) 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 Branch Chief, or the Branch Chief's 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. Calculations for cyclohexane.

8.3. Data Sheet.

Attachment 8.2: Calculations for cyclohexane

Cyclohexane:

Molecular weight = 84.2 g/mol

1 ppm = 3.44 mg / m<sup>3</sup> @ 25 °C ; 760 mm Hg

1. Calculations for injection rate of cyclohexane.

$$\text{Injection rate (ml/hr)} = \frac{\text{Conc (ppm)} \times 3.44 \text{ ((mg / m}^3\text{)/ppm)} \times \text{Airflow (L/min)} \times 60 \text{ min}}{1000 \text{ (mg/g)} \times 1000 \text{ (L/m}^3\text{)} \times 0.7785 \text{ (g/ml)}}$$

	64 Lpm	100 Lpm
2600 ppm	44.12 ml/hr	68.93 ml/hr

2. Calculations for actual challenge concentration.

$$\text{Conc (ppm)} = \frac{\text{amount delivered (g)} \times 24.45 \text{ (L/mol)}}{\text{Airflow (Lpm)} \times \text{Test time (min)} \times 84.2 \text{ (g/mol)}}$$

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Attachment 8.3: Test Data Sheet

### Revision History

<b>Revision</b>	<b>Date</b>	<b>Reason for Revision</b>
1.0	20 September 2004	Historic document
1.1	29 June 2005	Update header and format. No changes to method