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National Personal Protective Technology Laboratory
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Procedure No. CET-APRS-CBRN-STP-0410	Revision: 1.1	Date: 21 December 2005
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DETERMINATION OF CBRN FORMALDEHYDE 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 Formaldehyde 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, 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 Escape Respirator (APER)* Dated September 30, 2003.

2. GENERAL

This STP describes the *Determination of CBRN Formaldehyde 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 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 Lpm +/- 2%), and relative humidity (10-98 +/- 3%) and temperature (20-30 °C +/- 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°C to $130^{\circ}\text{C} \pm 0.2^{\circ}\text{C}$; relative humidity is 1% to $95\% \pm 0.5\%$
- 3.1.3. 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.
- 3.1.4. Interscan Corporation, Model RM-16-2, formaldehyde detector or equivalent. See Picture 3. Used for detection of breakthrough concentration. Detector range: 0 - 1999 ppb, resolution: 1 ppb.
- 3.1.5. Dry Gas Meter. Must have NIST traceable calibration certificate.
- 3.1.6. Electronic balance with accuracy of 0.01 grams (g).
- 3.1.7. Permeation Tubes of formaldehyde. Used for the calibration of both high and low range formaldehyde detectors.
- 3.1.8. Miller Nelson Research, Model SV-2000, Vaporizer system or equivalent. Used for vaporizing formaldehyde solution.
- 3.1.9. 40 Liter glass jug. Used for mixing of formaldehyde vapor.
- 3.1.10. Gilibrator, Primary Standard Airflow Calibrator or equivalent.
- 3.1.11. Gilian Gil-Air-3 Sampling Pump, or equivalent.
- 3.1.12. Graduated impinger, 25ml.
- 3.1.13. Para-formaldehyde, purified grade.
- 3.1.14. Sodium Thiosulfate (granular) or 0.1N certified sodium thiosulfate solution.
- 3.1.15. Starch, Soluble Potato, Powder.
- 3.1.16. Iodine, 0.1N certified solution.
- 3.1.17. Sodium Bisulfite, granular.
- 3.1.18. Sodium Carbonate, anhydrous powder.

3.1.19. Glacial acetic acid.

3.1.20. Heated magnetic stirrer and stir bar.

3.2 Test fixture for mounting canister.

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 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 (APER) 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 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 Prepare solutions (must be made at least 24 hours in advance)
- 5.1.1 12.5 % Paraformaldehyde solution: In a 2000 ml beaker containing 1000 ml distilled water and a stirring bar, add 125 g paraformaldehyde. Stir and heat the solution to 80°C using the heated magnetic stirrer. Place a watch glass over the top of the beaker or cover with aluminum foil to prevent excessive water loss. Continue stirring and heating until the solution clarifies (about 24 hours). Filter the solution if not clear after 24 hours. Store in covered bottle or flask.
- 5.2. Follow the 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.
- 5.3. After the manufacturer's specified warm-up period, calibrate the formaldehyde analyzers using a Kin-Tec generator and permeation tubes to generate approximately 1 ppm.

- 5.4. Set the high precision pump for delivery of calculated formaldehyde to obtain 250 ppm for a general test category or 500 ppm for a specific test category at the testing airflow. See appendix 8.2.
- 5.5. Set up test equipment as shown in Figure 1 or Figure 2 depending on the test parameters in section 6.3.
 - 5.5.1 For section 6.3.2 test: The airflow is controlled by the Mass Flow Controller. Humidity and temperature readings are monitored by a 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.
 - 5.5.2 For section 6.3.3 and 6.3.4 tests: The airflow, humidity and temperature are controlled by the Miller Nelson system. The temperature and humidity is monitored a 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 Air and water (if needed) supplies.
 - 5.6.2 Miller Nelson Research Flow-Temperature-Humidity Control System.
 - 5.6.3 Miller Nelson Research Vaporizer system.
 - 5.6.4 Amersham Biosciences High Precision Pump.
- 5.7. Set the airflow to the required airflow for the test. Verify the airflow from the test fixture using the appropriate dry test meter.
- 5.8. Establish the correct humidity and temperature depending on the test parameters in section 6.3.
 - 5.8.1 Fill pump reservoir with distilled H₂O. Insert needle from pump to top of column. Start the high precision pump flow using the calculated value from section 5.3. Allow 15 minutes for equalization system. Make sure distilled H₂O is delivered to the heated vaporizing element.
 - 5.8.2 Determine the humidity readings for distilled H₂O.

- 5.8.2.1 For section 6.3.2, the humidity value should be reading according to the specification required ($25 \% \pm 5\%$). If the humidity readings are not within the specification, make adjustments to the incoming compressed air or the room temperature to establish the specification.
- 5.8.2.2 For section 6.3.3 and 6.3.4, the humidity readings can be adjusted using the Miller Nelson Controller to the specification required ($50 \% \pm 5\%$ or $80\% \pm 5\%$).
- 5.9 Fill pump reservoir with 12.5 % paraformaldehyde solution, weigh and record weight of the reservoir. Insert needle from pump to top of column. Start the high precision pump flow. Allow 30-40 minutes for equalization of concentration. Make sure formaldehyde is delivered to the heated vaporizing element.
- 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 and place in testing chamber.
- 5.14 Determine the concentration of the formaldehyde vapor being generated.
- 5.14.1 Add 10 ml 1% sodium bisulfite solution to two impingers. Connect the two impingers together from outlet of one impinger to inlet of second impinger.
- 5.14.2 Attach Gil-Air 3 sampling pump to intake side of the first impinger. Connect outlet side of second impinger to Gilibrator. Check 1 Lpm flow of the pump pulling through the sodium bisulfite solution. This setting will be used to sample the formaldehyde concentration. Remove outlet side from Gilibrator.
- 5.14.3 Connect tubing from the sample side of the first impinger into the Gil-Air pump and tubing from the inlet side of the second impinger to the test concentration.
- 5.14.4 Turn on sampling pump and sample for 30 minutes at 1 Lpm.

- 5.14.5 Shut off sampling pump and transfer the contents of the two impingers into an Erlenmeyer flask. Rinse the impinger stem and body and transfer the washings to the flask.
- 5.14.6 Place a stir bar in the flask and place the flask on the magnetic stirrer. Begin stirring.
- 5.14.7 Add 1 ml starch solution.
- 5.14.8 Add 0.1N Iodine dropwise until solution is dark blue.
- 5.14.9 Add 0.1N sodium thiosulfate dropwise until blue color is gone indicating neutralization of the excess iodine.
- 5.14.10 Slowly add 0.01N iodine dropwise from a 50 ml buret until the first appearance of a faint blue color.
- 5.14.11 Add 25 ml sodium carbonate buffer solution. The blue color should disappear if formaldehyde is present.
- 5.14.12 Record the initial buret reading of the 0.01N Iodine.
- 5.14.13 Slowly add the 0.01N Iodine until the appearance of a faint blue color.
- 5.14.14 Record the final buret reading.
- 5.14.15 Calculate the concentration of formaldehyde using the formula in Attachments 8.3. Adjust the flow from the high precision pump and repeat steps 5.12 through 5.26 if concentration of $500 \text{ ppm} \pm 10\%$ is not obtained. If the concentration is correct attach sample line from high range detector to the tee in the system. This detector will be used to monitor the challenge concentration throughout the length of the test. Turn on the strip chart recorder to record the concentration.
- 5.14.16 Once the formaldehyde concentration has been established, testing may begin.
- 5.14.17 Direct challenge concentration airflow into test chamber.
- 5.14.18 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.

5.14.19 Run test until breakthrough of 10 ppm is observed or minimum service life is surpassed. Record this data on the test data sheet.

5.14.20 Weigh and record on the test data sheet the final weight of test canister.

5.14.21 Repeat steps 5.7 through 5.20 for each test described in section 6.3.

5.14.22 Turn off pump and heating mantel.

5.14.23 Allow clean air 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); 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. FORMALDEHYDE 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 250 ppm formaldehyde. Minimum service life will be 15, 30, 45, 60 or 90 minutes as per manufacturer 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 250 ppm formaldehyde. Minimum service life will be 15, 30, 45, 60 or 90 minutes as per manufacturer 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 250 ppm formaldehyde. Minimum service life for all canisters must be 5 minutes.

6.3.1.5. End of service life concentration is 10 ppm formaldehyde.

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 500 ppm formaldehyde. Minimum service life will be 15, 30, 45, 60 or 90 minutes as per manufacturer 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 500 ppm formaldehyde. Minimum service life will be 15, 30, 45, 60 or 90 minutes as per manufacturer 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 500 ppm formaldehyde. Minimum service life for all canisters must be 5 minutes.

6.3.2.5. End of service life concentration is 10 ppm formaldehyde.

7. RECORDS AND TEST SHEETS

7.1. All test data will be recorded on the FORMALDEHYDE 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 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 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 Figure 1- Bench Top Set-Up.

8.2 Calculations for formaldehyde delivery.

8.3 Data Sheet.

8.4 Qualitative analysis and calculations for determining concentration of challenge formaldehyde vapor.

Attachments 8.2: Calculations for formaldehyde delivery

Calculations for formaldehyde concentration.

$$1 \text{ ppm} = 1.23 \text{ mg} / \text{m}^3$$

$$\text{Airflow}_{(L/hr)} = \text{Airflow}_{(L/min)} \times 60 \text{ min}$$

$$\text{Injection rate (ml/hr)} = \frac{\text{Conc (ppm)} \times 30 \text{ (g / mol)} \times \text{Airflow (L/hr)}}{22.4 \times 10^6 \times 1.04 \text{ g/ml} \times 12.5\%}$$

	64 Lpm	100 Lpm
250 ppm	10 ml/hr	15 ml/hr
500 ppm	20 ml/hr	30 ml/hr

Attachment 8.4: Qualitative analysis and calculations for determining concentration of challenge formaldehyde vapor

CALCULATION OF FORMALDEHYDE CONCENTRATION

The formaldehyde equivalent F_e is given by the formula:

$$F_e = \left(\frac{(N) I}{.01} \right) \times 0.15 \text{ mg.}$$

Where (N) I is the normality of the standardized iodine. This formula is based on the fact that the equivalent weight of formaldehyde is 15 gm., which means that 1 ml. of 0.01N iodine is equivalent to 0.15 mg. of formaldehyde.

The amount of formaldehyde in the sample volume collected is given by the formula:

$$F_c = \text{final buret reading} - \text{initial buret reading} \times F_e \text{ mg}$$

$$F_c = \text{total ml. .01 iodine used} \times 0.15 \text{ mg}$$

Formaldehyde concentration in ppm is given by the following formula:

$$\text{ppm} = \frac{F_c}{V} \times \frac{24.45}{MW} \times \frac{760}{P} \times \frac{T+273}{298} \times 10^3$$

Where:

- V = volume of sample collected (30 lpm)
- MW = molecular weight formaldehyde (30.03 g/mole)
- P = pressure in mm of mercury in the lab
- T = temperature in °C of the lab

Assuming that T is equal to 25°C and p is equal to 760 mm mercury, the ppm formula simplifies to:

$$\text{HCHO (ppm)} = F_c \times 27.14 \text{ assuming volume of sample collected 30 liters}$$

$$\text{HCHO (ppm)} = \text{ml. 0.01 Iodine used} \times 0.15 \text{ mg} \times 27.14$$

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