National Institute for Occupational Safety and Health National Personal Protective Technology Laboratory P.O. Box 18070 Pittsburgh, PA 15236		
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DETERMINATION OF HYDROGEN FLUORIDE SERVICE LIFE TEST,
AIR-PURIFYING RESPIRATORS
STANDARD TESTING PROCEDURE (STP)

1. PURPOSE

This test establishes the procedure for ensuring that the level of protection provided by the hydrogen fluoride service life requirements on chemical cartridges air-purifying respirators 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), Subpart L, Section 84.190(b), and Subpart KK, Section 84.1157; Volume 60, Number 110, June 8, 1995.

2. GENERAL

This STP describes the Determination of Hydrogen Fluoride Service Life Test, Air-Purifying 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/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.

3.1.2. CEA Instruments Hydrogen Fluoride Analyzer Model-TG-700BA or equivalent.

3.1.3. Three-necked round bottom flask (250ml).

3.1.4. Heated vaporizing element to suspend in the three necked round bottom flask. The element is covered with platinum foil. The hydrogen fluoride is delivered via the syringe and tubing assembly to the element, where it is vaporized into the air-stream. This is not commercially available.

3.1.5. Vaisala model HMI 31 humidity indicator.

3.1.6. Hydrofluoric acid, 48% purity.

Approvals:	1st Level	2nd Level	3rd Level

3.1.7. Plastic syringe, 1 ml with Hamilton Teflon Tubing Assembly 90628.

3.1.8. Electronic balance with accuracy of 0.001 grams (g).

3.1.9. Sage syringe pump, Model 355 or equivalent.

3.2. Test fixture for mounting cartridges. The test fixture used is specific to each manufacturer depending on how the cartridge, canister, or powered air-purifying respirator (PAPR), mouth bit, etc. is mounted to the facepiece. The T-end has a 29/42 ground glass joint glued in place. Canisters are tested with their connections glued into the ground glass joint. In most cases the cartridge cups of the respirator are affixed by hot melt glue to PVC pipe tee of appropriate size. PAPR cartridges and canisters are tested on their blower units if possible, or on suitable substitutions, if the unit is too large for the test chamber.

3.3. The test chamber consisting of a 12" x 11½" x 7" air tight box, made of ½" plexiglass with 2 hinge type locks on the door opening lined with gasket material. A ½" hole is located on the back of the test chamber for the introduction of the test concentration and a 1½" hole on the top for the exit of the test fixture and to detect the breakthrough concentration. This fixture is not commercially available.

3.4. Resistance tester consisting of a vacuum source capable of delivering 85 liters per minute (lpm), a 6-inch slant manometer, and a 29/42 female ground glass joint. The resistance testers currently being used are located on the silica dust chamber.

4. TESTING REQUIREMENTS AND CONDITIONS

4.1. 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).

4.2. Normal laboratory safety practices must be observed. This includes safety precautions described in the current ALOSH Facility Laboratory Safety Manual.

4.2.1. Safety glasses, lab coats, and hard-toe shoes must be worn at all times.

4.2.2. Work benches must be maintained free of clutter and non-essential test equipment.

4.2.3. When handling any glass laboratory equipment, lab technicians and personnel must wear special gloves which protect against lacerations or punctures.

4.3. HYDROGEN FLUORIDE BENCH TEST FOR CARTRIDGES

4.3.1. Resistance to air flow will be taken before and after each test (see 84.203).

4.3.2. Three "as received" cartridges (or pairs of cartridges) will be tested at 64 lpm, continuous air flow, 50 ± 5 percent relative humidity (RH), approximately 25 degrees Celsius (^{O}C), and 70 ppm HF.

4.3.3. Two cartridges or pairs of cartridges will be equilibrated at room temperature by passing 25 percent RH air through them at 25 lpm for 6 hours and then testing them at 25 percent RH, approximately 25^{O}C, and 64 lpm continuous air flow rate containing 70 ppm HF.

4.3.4. Two cartridges or pairs of cartridges will be equilibrated at room temperature by passing 85 percent RH air through them at 25 lpm for 6 hours and then testing them at 85 percent RH, approximately 25^{o}C, and 64 lpm continuous air flow rate containing 70 ppm HF.

4.4. HYDROGEN FLUORIDE BENCH TESTS FOR PAPR CARTRIDGES

4.4.1. Resistances and airflows for tight fitting PAPR will be taken before and after each test. Airflows only for loose fitting PAPR will be taken before and after each test.

4.4.2. Three cartridges (or pairs of cartridges) will be tested at 115 lpm for tight fitting PAPR or 170 lpm for loose fitting PAPR hood or helmet continuous air flow, approximately 25^{o}C, and 50 ± 5 percent RH, approximately 25 degrees Celsius (^{O}C), and 70 ppm HF.

4.4.3. Two cartridges (or pairs of cartridges) will be equilibrated by passing 115 lpm for tight fitting PAPR or 170 lpm for loose fitting PAPR hood or helmet, continuous air flow through them at approximately 25^{o}C, and 25 percent RH for six hours. They will then be tested at 115 or 170 lpm continuous air flow, approximately 25^{o}C, 50 ± 5 percent RH, approximately 25 degrees Celsius (^{O}C), and 70 ppm HF.

4.4.4. Two cartridges (or pairs of cartridges) will be equilibrated by passing 115 lpm for tight fitting PAPR or 170 lpm for PAPR loose fitting hood or helmet, continuous air flow through them at 25^{o}C, and 85 percent RH for six hours. They will then be tested at 115 or 170 lpm continuous air flow, 25^{o}C, 50 ± 5 percent RH, approximately 25 degrees Celsius (^{O}C), and 70 ppm HF.

4.5 **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 warmup period, calibrate the HF analyzer following the instruction manual.

5.3. Set up test equipment as shown in Figure 1 for cartridge testing or Figure 2 for canister testing. In addition to the humidity reading controlled by the Miller Nelson system, the Vaisala HMI 31 humidity indicator sensor is inserted into the air stream via a tee set-up directly prior to the introduction of the hydrogen fluoride. This set up is not shown on the set ups. The humidity reading obtained at this point takes into account tubing length and outside hood air temperature.

5.4. Turn on:

5.4.1. Miller Nelson Unit.

5.4.2. Air and water supplies.

5.5. For cartridge testing at 70 ppm, fill syringe with hydrogen fluoride and place on syringe pump.

5.6. Establish the test concentration.

5.7. Determine the rate of advance of the syringe pump for the appropriate syringe volume, airflow, and the desired concentration for the respirator being tested. (See Tables 1 and 2.) Concentration will vary with the syringe volume size. The rate of the syringe pump must be recalculated when different size syringes are used or the airflow rate or concentration changes. For a concentration of 70 ppm HF, a flow rate of 0.0065 ml/min is required.

5.8. Allow the concentration to stabilize for 20-30 minutes.

5.9. Weigh the cartridge and record the weight.

5.10. Take inhalation and exhalation resistances of the cartridge mounted on the facepiece at 85 lpm. See Sections 84.203, 84.1157, Title 42, Code of Federal Regulations, Part 84 for breathing resistance requirements. Take airflows of PAPR cartridge mounted on blower assembly.

5.11. Mount cartridge onto test fixture and place in testing chamber.

5.12. Direct challenge concentration airflow into test chamber. Start timer. Mount small piece of tygon tubing onto the outlet of the test fixture. Insert intake tubing of detector into a slit cut into the side wall of the tubing to allow the detector to sample at the flow rate of the detector without interference from airflow back pressure. Monitor and record

upstream and downstream temperatures throughout testing. Record breakthrough values and times.

5.13. Run test until breakthrough of 3.0 ppm is observed or minimum service life is surpassed depending on type of cartridge or canister tested.

5.14. Dismount cartridge and weigh and record final weight, and take final inhalation and exhalation resistances and PAPR airflows.

5.15. Shut off syringe pump.

5.16. Disconnect air line from Miller Nelson system to flask to prevent contamination the humidity sensor.

5.17. Allow clean air to purge through system for 10 - 15 minutes.

5.18. Turn off air and water supply to Miller Nelson system.

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), Subpart L, Section 84.190(b), 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.

84.190 Chemical cartridge respirators: description.

(b) Chemical cartridge respirators for respiratory protection against gases or vapors, which are not specifically listed with their maximum use concentration, may be approved if the applicant submits a request for such approval, in writing, to the Institute. The Institute shall consider each such application and accept or reject the application after a

review of the effects on the wearer's health and safety and in the light of any field experience in use of chemical cartridge respirators as protection against such hazards.

84.1157 Chemical cartridge respirators with particulate filters; performance requirements; general. Chemical cartridge respirators with particulate filters and the individual components of each such device shall, as appropriate, meet the following minimum requirements for performance and protection:

(a) Breathing resistance test. (1) Resistance to airflow will be measured in the facepiece, mouthpiece, hood, or helmet of a chemical cartridge respirator mounted on a test fixture with air flowing at a continuous rate of 85 liters per minute, both before and after each test conducted in accordance with paragraphs (d) through (f) of this section

(2) The maximum allowable resistance requirements for chemical cartridge respirators are as follows:

Maximum Resistance
[mm. water-column height]

Type of chemical cartridge respirator	Inhalation		Exhalation
		Final[1]	
For gases, vapors, or gases and vapors, and dusts, fumes, and mists	50	70	20
For gases, vapors, or gases and vapors, and mists of paints, lacquers, and enamels	50	70	20

[1]Measured at end of service life specified in Table 11 in subpart L of this part.

(b) Facepiece test. The facepiece test will be conducted as specified in §84.205.

(f) Bench tests; gas and vapor tests.

(1) Bench tests will be made in accordance with 84.207 and tested cartridges shall meet the minimum requirements set forth in Table 11 of Subpart L of this part. Cartridges will be equilibrated in accordance with paragraph (f)(2) of this section.

(2)(i) Two powered air-purifying cartridges or pairs of cartridges will be equilibrated at room temperature by passing 25 percent RH air through them at the following flow rates (expressed in liters per minute (l.p.m.)) for 6 hours:

Type of cartridge	Airflow rate, l.p.m.
Powered air purifying with tight-fitting facepiece	115
Powered air purifying with loose-fitting hood or helmet	170

(ii) Two powered air-purifying cartridges or pairs of cartridges will be equilibrated by passing 85 percent relative humidity air through them at the flow rates stated in paragraph (f)(2)(i) of this section.

(iii) All cartridges will be resealed, kept in an upright position, at room temperatures, and tested within 18 hours.

6.3. The criterion for passing this test is that the penetration of HF shall not exceed 3 ppm for a minimum service life of 30 minutes for negative pressure cartridges and powered air-purifying cartridges. If the penetration does not exceed 3 ppm during the service life of 30 minutes for negative pressure cartridges and 30 minutes for powered air-purifying cartridges, the units pass the test. If the penetration does exceed 3 ppm during the service life of 30 minutes for negative pressure cartridges and 30 minutes for powered air-purifying cartridges, the units fail the test. The units will be run until the penetration of HF is equal to 3 ppm or the minimum service life is surpassed.

7. RECORDS/TEST SHEETS

7.1. All test data will be recorded on the HYDROGEN FLUORIDE 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 Table 1- Calculation for syringe pump injection rates.

8.2 Table 2 - Nominal injection rates for Sage Model 355.

8.3. Bench Top Set-Up.

8.4 Data Sheet.

TABLE 1

CALCULATIONS FOR SYRINGE PUMP INJECTION RATES at 25°C and 1 atm

For liquids:

$$C = \frac{(24.6 \times 10^6)\ KRp}{QM}$$

$$R = \frac{CQM}{(24.6 \times 10^6)\ KpX}$$

where:

R= rate of advance (mm/min) C= concentration (ppm)
K= syringe constant (ml/mm) p= solvent density (g/ml)
Q= airflow rate (lpm) M= molecular weight (g/mol)
X= decimal percent hydrogen fluoride solution

To obtain the syringe constant, divide the volume of the syringe in ml by the length of the syringe in mm.

Sample calculation: Find the rate of advance and % of flow setting required to produce a concentration of 70 ppm HF in 64 lpm of air, using a 1 ml syringe and a 48 percent paraformaldehyde solution.

$$R = \frac{(70\ ppm)\ (64\ lpm)\ (20.0 g/mol)}{(24.6 \times 10^6)\ (0.0172\ ml/mm)\ (1.0\ g/ml)(0.048)}$$

R= 0.382 mm/min.

From Table 2, the flow setting would be 264 at a range setting of 1/100.

For syringe pumps delivering in volumes of ml/minute, multiply rate of advance R by syringe constant.

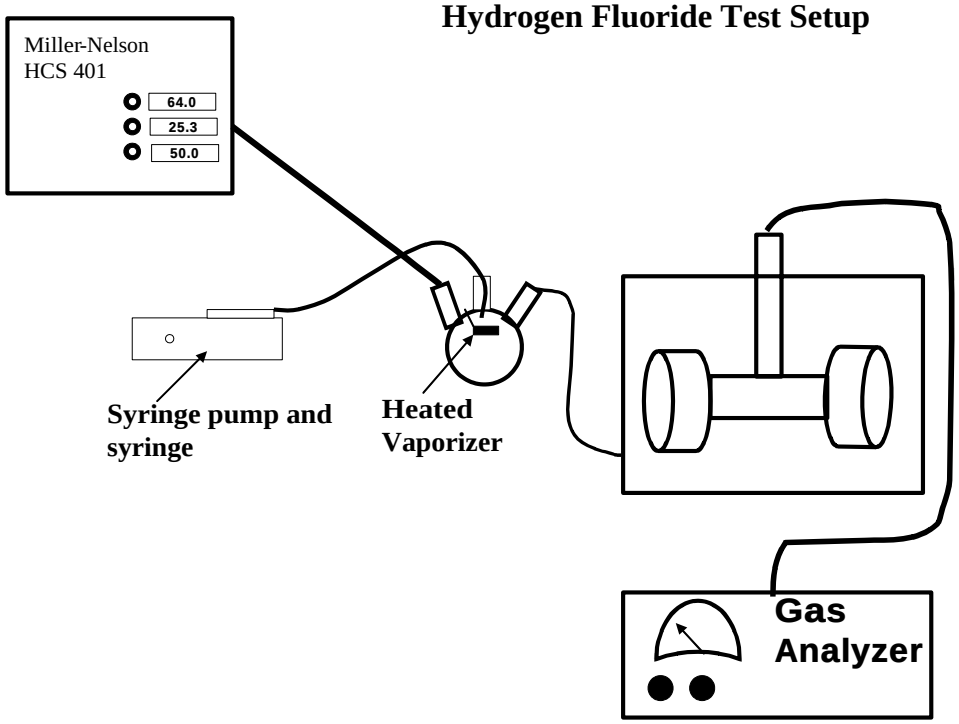
ml/min.= 0.382 mm/min. x 0.0172 ml/mm.
ml/min.= 0.0065

TABLE 2

NOMINAL INJECTION RATES FOR SAGE SYRINGE PUMP MODEL 355

	Rate of advance (R) (mm/min.)			
% Flow dial setting	Range setting 1	1/10	1/100	1/1000
100	14.5	1.45	0.145	0.0145
150	21.8	2.18	0.218	0.0218
200	29.0	2.90	0.0290	0.0290
250	36.3	3.63	0.0363	0.0363
300	43.5	4.35	0.0435	0.0435
350	50.8	5.08	0.508	0.0508
400	58.0	5.80	0.580	0.0580
450	65.3	6.53	0.653	0.0653
500	72.5	7.25	0.725	0.0725
550	79.8	7.98	0.798	0.0798
600	87.0	8.70	0.870	0.0870
650	94.3	9.43	0.943	0.0943
700	102	10.2	1.02	0.102
750	109	10.9	1.09	0.109
800	116	11.6	1.16	0.116
850	123	12.3	1.23	0.123
900	131	13.1	1.31	0.131
950	138	13.8	1.38	0.138
1000	145	14.5	1.45	0.145

Hydrogen Fluoride Test Setup



RB - RESPIRATOR CERTIFICATION TEAM

GAS & VAPOR RESPIRATOR TEST DATA SHEET (Ref.33-48,50,62) **STP No.: [_____]**

Task Number: TN-_____ Gas Name:

Manufacturer: _____ Item Tested:

RESISTANCE	Maximum Allowable Resistance (mm of H_2O)				Actual Resistance (mm of H_2O)				Result
Test	Inhalation		Exhalation		Inhalation		Exhalation		
			Initial		Initial	Final	Initial	Final	
1									
2									
3									
4									
5									
6									
7									

Overall Results: Pass ___ Fail ___ Comment:

WEIGHTS AND AIRFLOWS	**WEIGHTS (gm)**					**AIRFLOW (lpm)**			
		Con'd			**Conc.**	**Test Rate**		**(PAPR Only)**	
Test					**(ppm)**	RH%	lpm	Initial	Final
1									
2									
3									
4									
5									
6									
7									

Overall Results: Pass ___ Fail ___ Comment:

DATA TABLE	**Test Cond.**	**Final Time (min)**	**Leakage (ppm)**	**Temperature (ºC)**		**Corrected Time (min)**
Test				**Dnstream**	**Upstream**	
1						
2						
3						
4						
5						
6						
7						

Overall Results: Pass ___ Fail ___ Comment:
Was all testing equipment in calibration throughout all testing: Yes __ No
Signature: _____ Date:

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GAS & VAPOR RESPIRATOR TEST DATA SHEET (Ref.33-48,50,62) **STP No.: [_____]**

Task Number: TN-_____ Gas Name:
Manufacturer: _____ Item Tested:

Additional Comments:

Signature: _____ Date:

Revision History

Revision	Date	Reason for Revision
1.0	13 March 2002	Historic document
1.1	29 June 2005	Update header and format to reflect lab move from Morgantown, WV No changes to method