National Institute for Occupational Safety and Health National Personal Protective Technology Laboratory P.O. Box 18070 Pittsburgh, PA 15236		
Procedure No. RCT-APR-STP-0047	Revision: 1.1	Date: 24 August 2005

DETERMINATION OF PHOSPHINE 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 phosphine service life requirements on gas mask 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) and Subpart I, Section 84.110(c); Volume 60, Number 110, June 8, 1995.

2. GENERAL

This STP describes the Determination of Phosphine Service Life Test, Air-Purifying Respirator 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 Model TG-4000BA Phosphine detector or equivalent.

3.1.3. Tylan Flow Controller, model RO14

3.1.4. Tylan mass flowmeter, 5 lpm, 2 lpm or equivalent.

3.1.5. Certified cylinder of 1 parts per million (ppm) PH_3 in Nitrogen.

3.1.6. Gilian Gil-Air-3 Sampling Pump, or equivalent.

3.1.7. Vaisala model HMI 31 humidity indicator.

Approvals:	1st Level	2nd Level	3rd Level

3.1.8. Phosphine cylinder, 99% purity.

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

3.1.10. Nitrogen cylinder, 99% purity.

3.1.11. 4-way Purge regulator for phosphine cylinder.

3.2. Test fixture for mounting canisters. The test fixture used is specific to each manufacturer. Canisters are tested with their connections glued into the ground glass joint.

3.3. The test chamber consisting of a 12" x 11.5" 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 backside of the test chamber for the introduction of the test concentration and a 1.5" 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. PHOSPHINE BENCH TEST FOR ESCAPE GAS MASK CANISTERS

4.3.1. Resistance to air flow will be taken before and after each test, 84.126.

4.3.2. Three "as received" canisters (or pairs of canisters) will be tested at 64 Lpm, continuous air flow, 50 $\pm$ 5 percent relative humidity (RH), approximately 25 degrees Celsius (^{O}C), and 1500 ppm phosphine.

4.3.3. Two canisters or pairs of canisters will be equilibrated at room temperature by passing 25 percent RH air through them at 64 Lpm for 6 hours and then testing them at 25 percent relative humidity, approximately 25^{O}C, and 64 Lpm continuous air flow containing 1500 ppm phosphine.

4.3.4. Two canisters or pairs of canisters will be equilibrated at room temperature by passing 85 percent relative humidity air through them at 64 Lpm for 6 hours and then testing them at 85 percent relative humidity, approximately 25^{o}C, and 64 Lpm continuous air flow containing 1500 ppm phosphine.

4.4. PHOSPHINE BENCH TEST FOR TIGHT FITTING PAPR GAS MASK CANISTERS

4.4.1. Resistance to air flow will be taken before and after each test, 84.126.

4.4.2. Three "as received" canisters (or pairs of canisters) will be tested at 115 Lpm, continuous air flow, 50 $\pm$ 5 percent relative humidity (RH), approximately 25 degrees Celsius (^{O}C), and 1500 ppm phosphine.

4.4.3. Two canisters or pairs of canisters will be equilibrated at room temperature by passing 25 percent RH air through them at 115 Lpm for 6 hours and then testing them at 25 percent relative humidity, approximately 25^{O}C, and 115 Lpm continuous air flow containing 1500 ppm phosphine.

4.4.4. Two canisters or pairs of canisters will be equilibrated at room temperature by passing 85 percent relative humidity air through them at 115 Lpm for 6 hours and then testing them at 85 percent relative humidity, approximately 25^{o}C, and 115 Lpm continuous air flow containing 1500 ppm phosphine.

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 PH_3 analyzer using the certified gas cylinder containing the 1 ppm standard as follows:

5.2.1. With a tee in line on the gas cylinder, insert the intake tubing from the analyzer into the tee.

5.2.2. Turn on the 1.0 ppm certified phosphine cylinder.

5.2.3. Wait till the reading stabilizes, and adjust the span control to read 1.0 ppm.

5.3. Set up test equipment as shown in Figure 1. 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 gas. This set up is not shown on Figure 1. 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.

5.4.3. Phosphine cylinder.

5.4.4. Nitrogen cylinder.

5.5. Establish the test concentration for 1500 ppm Phosphine in 115 lpm air.

5.6. Calculate the rate of advance required to produce a concentration of 1500 ppm phosphine from Table 1. Determine the challenge test concentration via a dilution factor method by drawing off 1 Lpm test concentration and diluting with a known volume of clean air. By adjusting the rotameter and noting the deflection on the calibrated analyzer, the test concentration can be determined. The volume of clean air required to dilute the 1 Lpm of challenge concentration to produce 5000 ppm is calculated as follows:

5.6.1. $C_1V_1 = C_2V_2$ (Where C = concentration and V = volume)

5000 ppm x 1 Lpm = 50 ppm x V_2

$$V_2 = \frac{5000}{50}$$

$V_2 = 100$

Therefore: $V_2 - V_1 =$ volume of clean air

5.7. Draw off 1 Lpm of the phosphine in air test concentration and dilute it with the calculated volume of clean air for the required concentration.

5.8. Insert the intake tubing from the analyzer into the dilution air/gas stream. Adjust the rotameter till the analyzer reads 50 ppm. Note the position of the rotameter float for the concentration being determined.

5.8.1. As an example: A reading of 50 ppm for the dilution of 99 Lpm/1 Lpm phosphine test concentration results in a factor of 100. By using the following formula the total Phosphine test concentration can be determined.

Dilution factor x 50 ppm reading = Total Phosphine test concentration.

5.9. Once the Phosphine concentration has been established, testing may begin.

5.10. Weigh the canister and record the weight.

5.11. Take inhalation and exhalation resistances of the canister mounted on the facepiece at 85 Lpm. See Sections 84.122 Title 42, Code of Federal Regulations, Part 84 for breathing resistance requirements.

5.12. Mount canister onto test fixture and place in testing chamber.

5.13. 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.

5.14. Run test until breakthrough of 1.0 ppm is observed or minimum service life is surpassed.

5.15. Dismount canister, weigh and record final weight, and take final inhalation and exhalation resistances.

5.16. Shut off phosphine cylinder.

5.17. Disconnect phosphine tubing from the rotameter to prevent contamination of the

humidity sensor.

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

5.19. 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) 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.

84.110. Gas masks; description.

(c) Gas masks for respiratory protection against gases and vapors other than those specified in paragraph (b) of this section, may be approved upon submittal of an application in writing for approval to the Respirator Branch listing the gas or vapor and suggested maximum use concentration for the specific type of gas mask. The Institute will consider the application and accept or reject it on the basis of effect on the wearer's health and safety and any field experience in use of gas masks for such exposures. If the application is accepted, the Institute will test such masks in accordance with the requirements of this subpart.

7. RECORDS/TEST SHEETS

7.1. All test data will be recorded on the PHOSPHINE 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.

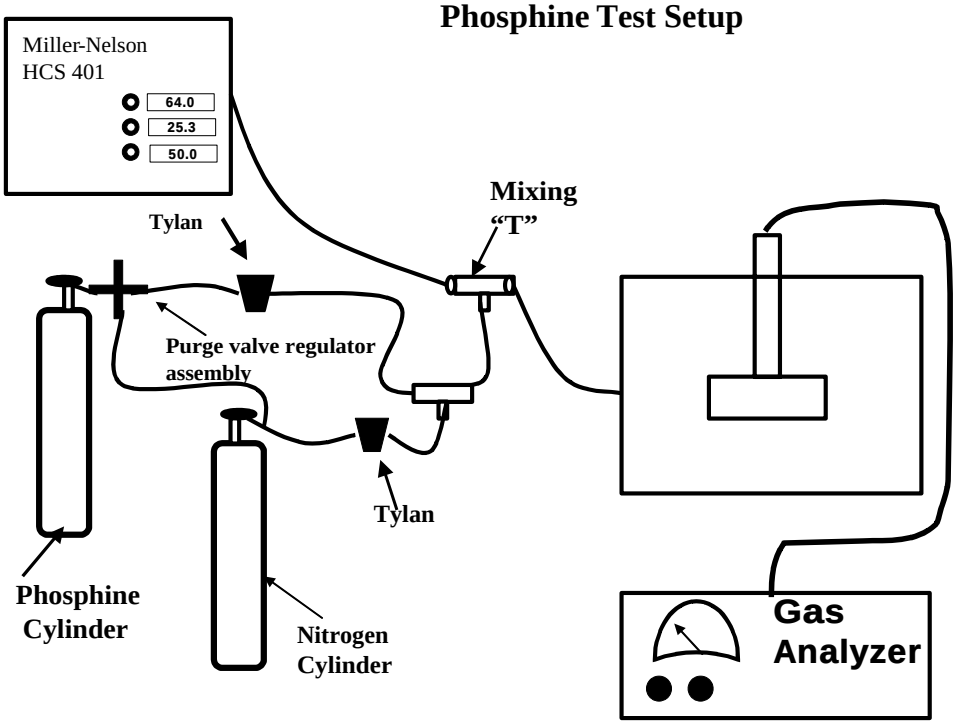
8. ATTACHMENTS

8.1. Bench Top Set-Up.

8.2. Data Sheet.

8.3. Table 1- Concentration Calculation for Syringe Pump Injection Rates.

Phosphine 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				**Dns tream**	**Upstr eam**	
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:

RB - RESPIRATOR CERTIFICATION TEAM ***Page 2***

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:

TABLE 1

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

For liquids:

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

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

For gases:

$$C = \frac{RK\ 10^3}{Q}$$

$$R = \frac{CQ}{K10^3}$$

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)

Sample calculation: Find the rate of advance required to produce a concentration of 1500 ppm phosphine gas in 115 lpm of air, using a 20 ml syringe constant.

$$R = \frac{(1500\ ppm)\ (115\ lpm)}{(.301\ ml/mm)\ 10^3}$$

R= 0.573 mm/min.

For calculating ml/min multiply rate in mm/minutes time the syringe constanst. syringe pumps delivering in volumes of ml/minute, multiply rate of advance R by syringe constant.

ml/min.= 0.573 mm/min. x 0.301 ml/mm.
ml/min.= 172.5

Revision History

Revision	Date	Reason for Revision
1.0	7 March 2002	Historic document
1.1	11 July 2001	Update header and format to reflect lab move from Morgantown, WV No changes to method