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 METHYLAMINE SERVICE LIFE TEST, AIR-PURIFYING RESPIRATORS WITH CANISTERS STANDARD TESTING PROCEDURE (STP)

1. PURPOSE

This test establishes the procedure for ensuring that the level of protection provided by air-purifying respirators with canisters (gas masks) submitted for Approval, Extension of Approval, or examined during Certified Product Audits, meet the minimum methylamine service life test requirements set forth in 42 CFR Part 84, Subpart I, Section 84.126.

2. GENERAL

This STP describes the Determination of Methylamine Service Life Test, Air-Purifying Respirators with canisters 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 / REFERENCES

3.1. The list of necessary test equipment and materials follows:

3.1.1. Miller Nelson Research Model 401 Flow-Temperature-Humidity Control System (250 lpm) or equivalent. Air flow control accuracy is ± 2% F.S. Temperature control accuracy is ± 1° C. Humidity control accuracy is ± 3% R.H.

3.1.2. Edge Tech Dew Prime II Hygrometer, Model 2000 or equivalent. Accuracy is ± 0.2° C, ±0.5% RH.

3.1.3. Radiometer America Multi-Titration System, Model DTS 800, burettes or equivalent.

3.1.4. Air-Sentry IMS Ammonia Analyzer Model 10R-NH3-4000M or equivalent. Range for ammonia is 0.1 to 50 ppmv ±0.1% of full scale. The analyzer may be used for methylamine due to the similar structure.

3.1.5. "The Gilibrator", Primary Standard Airflow Calibrator or equivalent.

3.1.6. Mass Flow Controllers, Brooks Instruments, variable flow rate depending on use, model series 5850S and 5853S. Accuracy is 0.7% setpoint & 0.2% FS.

Approvals: First Level	Second Level	Third Level	Fourth Level

3.1.7. Read Out and Control Electronics, Brooks Instruments, Model 0154.

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

3.1.9. Fisher Scientific Gas washing bottle or bubbler, catalog # 03-036 or equivalent.

3.1.10. American Meter Co. Dry Test Meter Models DTM-325 and DTM-200.

3.1.11. Erlenmeyer flasks or beakers, 250 to 500 milliliters (mL).

3.1.12. Certified cylinder of approximately 10 ppmv methylamine in nitrogen.

3.1.13. Pipettes, 5 ml.

3.1.14. Boric Acid (granular).

3.1.15. Bromophenol Blue (powder).

3.1.16. Sulfuric Acid (concentrated) or certified 0.025N sulfuric acid solution.

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

3.1.18. Methylamine cylinder, 99% purity.

3.2. Test fixture for mounting canisters. The test fixture used is specific to each manufacturer depending on how the canister is mounted to the facepiece. In most cases chin-style canisters use the 40 mm thread for which we have adapters. In cases where other thread sizes are used, the adapters of the respirator are affixed by hot melt glue to a PVC pipe tee of appropriate size. Front or back mounted canisters are tested with their breathing tubes and adapters.

3.3. The test chamber consisting of an approximately 12" x 12" x 7" air tight box, with 2 clamp type locks on the door opening lined with gasket material, and appropriate inlet, outlet and sampling ports. This fixture is not commercially available.

3.4. Refer to the following Work Instructions for further information on performing this test:
TEB-RCT-APR-WI-1009 – Laboratory Safety Procedures for Methylamine Tests
TEB-RCT-APR-WI-1109 – Calibration Procedures for Methylamine Tests
TEB-RCT-APR-WI-1209 – Start–Up and Shut–Down Procedures for Methylamine Tests
TEB-RCT-APR-WI-1309 – Using the LabView System for Methylamine Tests
TEB-RCT-APR-WI-1409 – Reporting Results for Methylamine Tests

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 testing laboratory's calibration procedure and schedule. All measuring equipment utilized for this testing must have been calibrated using a method traceable to the National Institute of Standards and Technology (NIST) when available.

4.2. 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.*
***Note** 4.2 does not apply to Pretest data from applicants as required under 42 CFR 84.64.

4.3. 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.

4.4. The precision and accuracy of this method was determined by validation testing of a single lot of commercially available multi-gas type cartridges. The results of these tests are shown in the table below.

TEST TYPE	MEAN SERVICE LIFE (MINUTES)	STD. DEV.
AS RECEIVED	79.34	2.37
EQUIL. 25% RH	198.32	10.27
EQUIL. 85% RH	248.36	22.69

4.5. Normal laboratory safety practices must be observed. Please refer to Material Safety Data Sheets and the current NIOSH Pittsburgh Health and Safety Program for the proper protection and care in handling, storing, and disposing of the chemicals and gases used in this procedure.

4.6. The cylinder of 99% methylamine, as well as the calibration gas cylinders, are typically used inside the laboratory fume hood. If there is a release of 99% methylamine outside the hood, sound an alarm, and any personnel in the laboratory should immediately exit from the building.

4.7. METHYLAMINE BENCH TEST FOR CANISTERS

4.7.1. Resistance to air flow of the complete respirator will be taken before and after each test (see 42 CFR 84.203). The standard testing procedures are described in TEB-APR-STP-003 and TEB-APR-STP-007.

4.7.2. Test conditions as required by 42 CFR 84.110 (c).

SAMPLE	CONDITION	EQUILIBRATION CONDITIONS FOR 6 HOURS			TEST CONDITIONS			CONCENTRATION PPMV METHYLAMINE CHIN STYLE AND ESCAPE	
		TEMP. ° C	AIRFLOW LPM	R.H. %	TEMP. ° C	AIRFLOW LPM	R.H. %	TEST	BREAKTHROUGH
1-3	AS RECEIVED	NA	NA	NA	25	64	50	5000	10
4-5	EQUIL. 25% R.H.	25	64	25	25	32	50	5000	10
6-7	EQUIL. 85% R.H.	25	64	85	25	32	50	5000	10

Tolerances:

PARAMETER	TOLERANCE
25°C	± 2.5°C
32 LPM	± 0.50 LPM
64 LPM	± 1.0 LPM
25% R.H.	± 3% R.H.
50% R.H.	± 3% R.H.
85% R.H.	+0/-5% R.H.
5000 ppmv	± 10%

NOTES: R.H. levels greater than 85% are difficult to maintain and may cause rapid degradation of service life.

Tolerance on accuracy of air flow rates exceeds specification on Miller Nelson control unit because flow rates are calibrated for every test. This improves the precision of the measurement and allows for the tighter tolerance on short-term drift.

4.7.3. All equilibrated canisters will be resealed, kept in a position such that the direction of airflow would be horizontal, at room temperature, and testing shall begin within 18 hours.

5. PROCEDURE

Note: Reference Section 3 for equipment, model numbers and manufacturers.
Work Instructions are to be used in conjunction with standard NIOSH test apparatus.

5.1. Set up the test equipment as shown in Figure 1.

5.2. Calibrate the breakthrough NH_3 analyzer using the certified gas cylinder containing the 10 ppmv methylamine standard.

5.3. Prepare solutions:

5.3.1. 5% boric acid: Heat, but do not boil, 50g boric acid in 1 liter distilled water.

5.3.2. 0.025N sulfuric acid solution: Weigh 1.28g concentrated sulfuric acid and add to a 1 liter flask containing 800 ml distilled water. Dilute to 1 liter mark with distilled water.

5.3.3. Bromophenol blue solution: Dissolve 0.5g bromophenol powder in 500 ml distilled water. Shelf life of this solution is one month.

5.4. Prepare comparison blank:

5.4.1. Measure 50 ml boric acid solution (100 ml for 30,000 ppmv) into 250 ml Erlenmeyer flask. Add 25 ml distilled water, 5 drops bromophenol blue indicator, and distilled water equal to the calculated volume of sulfuric acid needed to determine CH_2NH_3 concentration.

5.4.2. Example: for a comparison blank equal to 5000 ppmv.

$$\text{Calculated vol. } H_2SO_4 = \frac{\text{ppmv } CH_2NH_3}{\text{standard factor}}$$

Where the standard factor = 611 ppmv CH_2NH_3/ml H_2SO_4

$$\text{vol. } H_2SO_4 = \frac{5000}{611}$$

$$\text{vol. } H_2SO_4 = 8.2 \text{ ml}$$

Total volume of water added including the 25 ml from step 5.4.1. = 33.2 ml.

5.5. Establish the correct humidity and temperature for the sample being tested as per the test requirements in paragraph 4.7.

5.6. Set the airflow to the required level for the sample being tested as per the test requirements in paragraph 4.7 and the adjustments in paragraph 5.12. Calibrate the total airflow, including any additional flow arising from hygrometer flow rates, from the test fixture using the dry test meter.

5.7. Weigh the canister(s) and record the weight.

5.8. Measure initial inhalation and exhalation resistances of the canister(s) mounted on the facepiece as described in TEB-APR-STP-003 and TEB-APR-STP-007. Record values on the data sheet.

5.9. Make sure diverter valve in the system is diverting the challenge concentration airflow to discharge and not into the testing chamber.

5.10. Mount canister(s) onto test fixture and place in testing chamber.

5.11. Open the 99% methylamine cylinder.

5.12. Establish the test concentration of 5,000 ppmv ± 10% methylamine by setting the theoretical flow rate of pure methylamine to mix with the flow of air to produce the required concentration. Then, set the mass flowmeter to that level.

TOTAL FLOW RATE FOR TEST LPM	5000 PPMV TEST METHYLAMINE FLOW RATE CC/MIN
32	160
64	320

5.13. Measure 50 ml boric acid solution into the gas bubbler. Attach Gil-Air 3 sampling pump to the intake side of the gas bubbler. Connect outlet side of bubbler to Gilibrator. Calibrate 1 lpm ± 2% flow rate of the pump. This setting will be used to sample the methylamine upstream concentration.

5.14. Connect tubing from the sample side of gas bubbler into the Gil-Air pump and tubing from the inlet side of the gas bubbler into the upstream sampling port.

5.15. Turn Gil-Air pump on and sample at 1 lpm for 1 minute.

5.16. Remove gas bubbler, and transfer the solution into an Erlenmeyer flask.

5.17. Rinse the gas bubbler with 25 ml distilled water and transfer the washings into the flask.

5.18. Add 5 drops bromophenol blue indicator.

5.19. Titrate the sample with 0.025N H_2SO_4 until it matches the comparison blank color sample (see 5.3). Record ml used.

5.20. Calculate the concentration of CH_3NH_2 using the following formula:
Conc. in ppmv = ml H_2SO_4 used x standard factor of 611 ppmv CH_3NH_2/ ml H_2SO_4
Adjust the flowmeter setting as required. Once the methylamine concentration has been established and is stable, testing may begin. When approximately ½ of the required minimum service life has been reached, perform another titration to calculate the challenge concentration during the test.

5.21. Monitor and record challenge and breakthrough temperatures, challenge RH and breakthrough values and times throughout testing.

5.22. Run test until breakthrough of 10.0 ppmv is observed or minimum service life shown in section 6.2 is surpassed by 10%.

5.23. At end of test, system will automatically direct challenge concentration airflow through diverter valve to discharge.

5.24. Dismount canister(s), weigh and record final weight, and take final inhalation and exhalation resistances as described in TEB-APR-STP-003 and TEB-APR-STP-007. Measurement of the final inhalation and exhalation resistances is required for certification and audit testing.

5.25. If there is another sample to test, repeat steps 5.5 – 5.15.

5.26. After all tests are completed for the shift, set temperature and humidity to zero on the Miller Nelson system and allow clean air to pass through the system for 30 minutes. Purge the breakthrough and challenge detectors with clean air for 15 minutes.

6. PASS/FAIL CRITERIA

6.1. The legal basis for passing this test is set forth in 42 CFR Part 84, Subpart I, Section 84.110(c).

6.2. Minimum service life requirements for canisters are shown below.

Canister type	Test condition	Test atmosphere			Number of tests	Maximum allowable penetration (ppmv)[1]	Minimum service life (minutes)[2]
		Gas or vapor	Concentration (ppmv)	Flow rate (liters per minute)			
Methylamine Chin - Style	As received	CH_3NH_2	5,000	64	3	10	6/12
	Equilibrated	CH_3NH_2	5,000	32	4	10	6/12
Methylamine Escape	As received	CH_3NH_2	5,000	64	3	10	12
	Equilibrated	CH_3NH_2	5,000	32	4	10	12

[1]Minimum life will be determined at the indicated penetration.

[2]For a combination respirator designed for respiratory protection against one or more than one gas of a single type, as for use in ammonia and/ or methylamine, or a combination of more than one type of gas and vapor without carbon monoxide, the minimum life shall be 12 minutes. For a combination respirator designed for respiratory protection against one or more than one gas of a single type combined with carbon monoxide, or more than one type of gas or vapor, as for use in methylamine and in chlorine, combined with carbon monoxide, the minimum life shall be 6 minutes. For escape gas masks the minimum life shall be 12 minutes.

7. RECORDS/TEST SHEETS

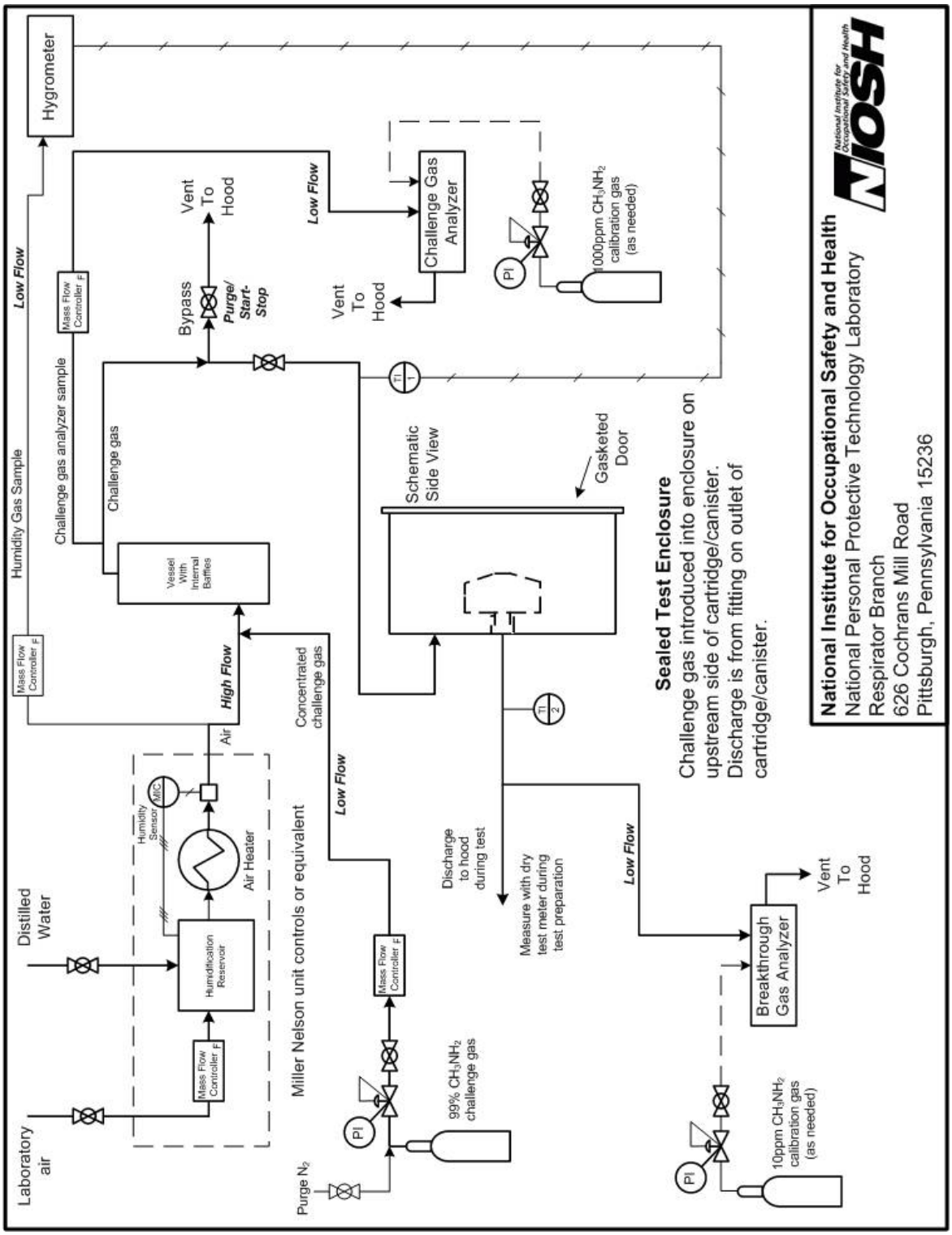
7.1. Record the test data in a format that shall be stored and retrievable.

8. ATTACHMENTS

8.1. Figure 1- Bench-Top Set-Up

8.2. Data Sheet

Figure 1- Bench-Top Set-Up



8.2. Data Sheet

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					**(ppmv)**	RH%	Lpm	Initial	Final
1									
2									
3									
4									
5									
6									
7									

Overall Results: Pass _____ Fail _____ Comment:

DATA TABLE	**Test Cond.**	**Final Time (min)**	**Leakage (ppmv)**	**Temperature (°C)**		**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:

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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	15 March 2002	Historic document
1.1	28 July 2005	Update header and format to reflect lab move from Morgantown, WV No changes to method
2.0	18 December 2008	Significant rewrite of RCT-APR-STP-0045. Changes affect form and provide clarification of technical content.