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National Personal Protective Technology Laboratory
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Procedure No. TEB-APR-STP-0048B	Revision: 2.0	Date: 1 December 2008

DETERMINATION OF SULFUR DIOXIDE 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 sulfur dioxide service life test requirements for acid gas canisters set forth in 42 CFR Part 84, Subpart I, Section 84.126.

2. GENERAL

This STP describes the Determination of Sulfur dioxide 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

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. Interscan Corporation Model RM-24-2 sulfur dioxide detector or equivalent. Detector range: 0 - 19.99 ppmv, resolution: 0.01 ppmv.

3.1.5. Interscan Corporation Model RM-24-0 sulfur dioxide detector or equivalent. Detector range: 0 - 1999 ppmv, resolution: 1 ppmv.

3.1.6. Interscan Corporation Model DS1 Dilution System or equivalent. Accuracy is ± 5% F.S. flowmeter reading.

Approvals: First Level	Second Level	Third Level	Fourth Level

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

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

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

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

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

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

3.1.13. Erlenmeyer flasks or beakers, 100 to 500 milliliters (ml)

3.1.14. Pipets, 5 ml.

3.1.15. Iodine (I_2) (crystals) or 0.025N certified iodine solution.

3.1.16. Potassium Iodide (KI) (granular).

3.1.17. Sodium Thiosulfate Pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$, crystalline) or 0.025 Normal (N) certified $Na_2S_2O_3$ solution.

3.1.18. Potassium Iodate (KIO_3) (granular).

3.1.19. Boric Acid (granular).

3.1.20. Starch, Soluble Potato, Powder.

3.1.21. Certified cylinder of approximately 5 ppmv sulfur dioxide in nitrogen.

3.1.22. Sulfur dioxide cylinder, 99 % purity.

3.1.23. Electronic balance with accuracy of 0.01 grams (g).

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-1008- Laboratory Safety Procedures for Sulfur dioxide Tests
TEB-RCT-APR-WI-1108- Calibration Procedures for Sulfur dioxide Tests
TEB-RCT-APR-WI-1208- Start–Up and Shut–Down Procedures for Sulfur dioxide Tests
TEB-RCT-APR-WI-1308- Using the LabView System for Sulfur dioxide Tests
TEB-RCT-APR-WI-1408 – Reporting Results for Sulfur dioxide 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 as a contractor 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	34.57	0.87
EQUIL. 25% RH	105.74	4.47
EQUIL. 85% RH	210.61	7.30

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

4.7. SULFUR DIOXIDE 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 84.122). 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 84.126.

SAMPLE	CONDITION	EQUILIBRATION CONDITIONS			TEST CONDITIONS			TEST CONCENTRATION		BREAKTHROUGH
		FOR 6 HOURS						FRONT AND BACK MOUNTED	CHIN STYLE AND ESCAPE	CONCENTRATION
		TEMP. ° C	FLOWRATE LPM	R.H. %	TEMP. ° C	FLOWRATE LPM	R.H. %	PPMV SO_2	PPMV SO_2	PPMV SO_2
1-3	AS RECEIVED	NA	NA	NA	25	64	50	20000	5000	5
4-5	EQUIL. 25% R.H.	25	64	25	25	32	50	20000	5000	5
6-7	EQUIL. 85% R.H.	25	64	85	25	32	50	20000	5000	5

Tolerances:

PARAMETER	TOLERANCE
25°C	± 2.5°C
32 LPM	± 0.5 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%
20,000 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. For calibration purposes use those described in the manufacturer's operation and maintenance manuals.

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

5.2. Calibrate the breakthrough SO_2 analyzer using the certified gas cylinder containing the 5 ppmv standard.

5.3. Prepare solutions. Commercially purchased certified solutions can be used.

5.3.1. 2 percent Potassium Iodide/1 percent Potassium Iodate ($KI0_3$): Dissolve 20 g potassium iodide and 10 g potassium iodate in 1 liter distilled water. Solution will be pale yellow. Cover flask with aluminum foil or store in light proof container. Discard when the solution is dark yellow in color.

5.3.2. 0.025N Sodium Thiosulfate Solution (0.025N $Na_2S_2O_3$): Dissolve 6.205 g sodium thiosulfate in 1 liter of distilled water.

5.3.3. Starch Indicator: Weigh 1g boric acid and add to 100 ml of distilled water, bring to a boil.

5.3.3.1. Weigh 1-2 g potato starch, add cold water to make a paste.

5.3.3.2. Add to boiling water and continue to boil for 1 minute.

5.3.3.3. Discard when starch solution becomes cloudy.

5.4. Measure 50 ml potassium iodate solution into a beaker. For higher concentrations use 100 ml potassium iodate. If solution is pale yellow, add a few drops of 0.025N $Na_2S_2O_3$ till the solution is colorless. Pour contents into a gas washing bottle. Attach the Gil-Air 3 sampling pump to intake side of the gas bubbler. Connect outlet side of bubbler to Gilibrator. Check 1 lpm flow of the pump pulling through the potassium iodate solution. This setting will be used to sample the sulfur dioxide concentration.

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. Calibrate the total airflow, including any additional flow arising from challenge gas flow rates and / or hygrometer flow rates, from the test fixture using the dry test meter.

5.7. Weigh the canister and record the weight.

5.8. Measure initial inhalation and exhalation resistances of the canister 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 onto test fixture and place in testing chamber.

5.11. Open the 99% sulfur dioxide cylinder.

5.12. Establish the test concentration of 5,000 ppmv or 20,000 ppmv ± 10% sulfur dioxide by setting the theoretical flow rate of pure sulfur dioxide to mix with the flow of air to produce the required concentration (see table below). Then, set the mass flowmeter to that level. Adjust the flowmeter setting as required. Once the sulfur dioxide concentration has been established by titration and/or dilution /measurement and is stable, testing may begin.

FLOW RATE FOR TEST	FLOW RATE OF PURE SULFUR DIOXIDE	
LPM	FOR 5000 PPMV	FOR 20000 PPMV
	sccm or mL/min.	
32	160	640
64	320	1280

5.13. 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 test gas concentration.

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

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

5.16. Rinse the gas bubbler with distilled water and transfer the washings into the flask.

5.17. Titrate the solution with 0.025N sodium thiosulfate until it is pale yellow.

5.18. Add 5 ml of starch indicator. Solution will turn dark blue.

5.19. Continue titration until the blue color just disappears. Record milliliters used.

5.20. Calculate the concentration of SO_2 in air using the following formula:

$$\text{Conc. (ppm)} = \text{ml of } 0.025\text{N } Na_2S_2O_3 \text{ x standard factor}$$

$$\text{Standard factor} = 305.8 \text{ ppm } SO_2 / \text{ml } Na_2S_2O_3$$

5.21. As an alternate or additional method to measure challenge concentration, a sample is diluted by a factor of 11:1 or other suitable factor using the dilution system and measured

on the detector used for challenge concentration. The dilution system is used because the test concentrations exceed the detector's upper limit. Once the test concentration has been established, testing may begin.

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

5.23. Run test until breakthrough of 5.0 ppmv is observed or minimum service life shown in section 6.2 is surpassed by 10%.

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

5.25. Dismount canister, 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.

5.26. If there is another sample to test, repeat steps 5.13 – 5.25.

5.27. 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 requirement for passing this test is set forth in 42 CFR Part 84 Subpart I, Section 84.126.

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

Canister type	Test condition	Test atmosphere			Number of tests	Maximum allowable penetration (parts per million volume)[1]	Minimum service life (minutes)[2]
		Gas or vapor	Concentration (parts per million volume)	Flow rate (liters per minute)			
Acid Gas[3] Front and Back Mounted	As received Equilibrated	SO_2 SO_2	20,000 20,000	64 32	3 4	5 5	6/12 6/12
Acid Gas[3] Chin-Style	As received Equilibrated	SO_2 SO_2	5,000 5,000	64 32	3 4	5 5	6/12 6/12
Acid Gas[3] Escape	As received Equilibrated	SO_2 SO_2	5,000 5,000	64 32	3 4	5 5	12 12

[1]Minimum life will be determined at the indicated penetration.
[2]For a combination front and back mounted or chin style gas mask canister respirator designed for respiratory protection against two or three of the following types: ammonia, acid gas, organic vapor, or carbon monoxide, the minimum life shall be 12 minutes. For a combination front and back mounted or chin style gas mask canister respirator designed for respirator protection against all of the following types: ammonia, acid gas, organic vapor, or carbon monoxide, the minimum life shall be 6 minutes each for ammonia, acid gas and organic vapor.
[3]For Acid Gas approval, canister must also pass chlorine testing.

7. RECORDS/TEST SHEETS

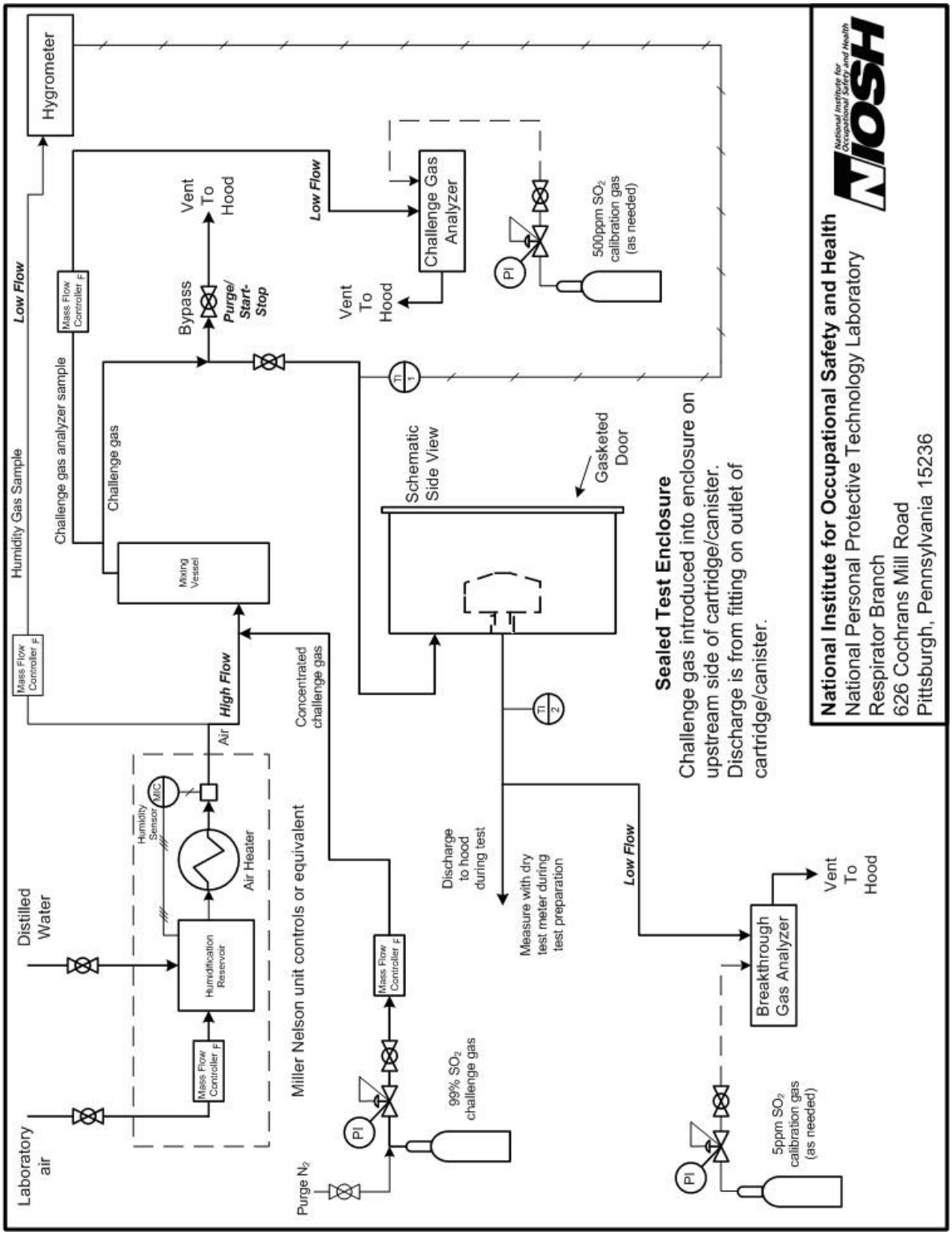
7.1. Record the test data in a format that shall be stored and retrievable.

8. ATTACHMENTS

8.1. Figure 1 - Bench-Top Set-Up

8.2. Data Sheet

8.1. 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 (g)**					**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	14 March 2002	Historic document
1.1	30 June 2005	Update header and format to reflect lab move from Morgantown, WV No changes to method
2.0	1 December 2008	Significant rewrite of RCT-APR-STP-0048. Changes affect form and provide clarification of technical content.