National Institute for Occupational Safety and Health
National Personal Protective Technology Laboratory
P.O. Box 18070
Pittsburgh, PA 15236

Procedure No. TEB-APR-STP-0043B	Revision: 2.0	Date: 14 March 2008

DETERMINATION OF HYDROGEN SULFIDE 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 hydrogen sulfide service life test requirements set forth in 42 CFR Part 84, Subpart G, Section 84.63 (a), (c), (d), and Subpart I, Section 84.110 (c).

2. GENERAL

This STP describes the Determination of Hydrogen Sulfide 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.

3.1.3. Interscan Corporation Model RM-17-2 hydrogen sulfide detector or equivalent. Detector range: 0 - 19.99 ppmv, resolution: 0.01 ppmv.

3.1.4. Interscan Corporation Model RM-17-0 hydrogen sulfide detector or equivalent. Detector range: 0 - 1999 ppmv, resolution: 1 ppmv.

Approvals:			
First Level	Second Level	Third Level	Fourth Level

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

3.1.6. 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.

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

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

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

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

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

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

3.1.13. Separatory funnel with rubber stopper, 125 ml.

3.1.14. Aspirator bulb with tubing and stopper.

3.1.15. Joint clamp.

3.1.16. Pipets, 10-20 ml.

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

3.1.18. Potassium Iodide (KI) (granular).

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

3.1.20. Sodium Hydroxide (NaOH) (granular).

3.1.21. Boric Acid (granular).

3.1.22. Starch, Soluble Potato, Powder.

3.1.23. Certified cylinder of approximately 10 ppmv hydrogen sulfide in nitrogen.

3.1.24. Hydrogen sulfide cylinder, 99 % purity.

3.1.25. 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-1006- Laboratory Safety Procedures for Hydrogen Sulfide Tests
TEB-RCT-APR-WI-1106- Calibration Procedures for Hydrogen Sulfide Tests
TEB-RCT-APR-WI-1206- Start–Up and Shut–Down Procedures for Hydrogen Sulfide Tests
TEB-RCT-APR-WI-1306- Using the LabView System for Hydrogen Sulfide Tests
TEB-RCT-APR-WI-1406 – Reporting Results for Hydrogen Sulfide 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	199.55	11.51
EQUIL. 25% RH	668.13	24.20
EQUIL. 85% RH	565.95	51.43

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% hydrogen sulfide, as well as the calibration gas cylinders, are typically used inside the laboratory fume hood. If there is a release of 99% hydrogen sulfide outside the hood, sound an alarm, and any personnel in the laboratory should immediately exit from the building. **THE LOWER EXPLOSIVE LIMIT OF H_2S IS 40,000 PPMV.**

4.7. HYDROGEN SULFIDE 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 84.63(a)(c)(d) and 84.110(c).

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 H2S	PPMV H2S	PPMV H2S
1-3	AS RECEIVED	NA	NA	NA	25	64	50	10000	5000	10
4-5	EQUIL. 25% R.H.	25	64	25	25	32	50	10000	5000	10
6-7	EQUIL. 85% R.H.	25	64	85	25	32	50	10000	5000	10

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%
10,000 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 H_2S analyzer using the certified gas cylinder containing the 10 ppmv standard.

5.3. Prepare solutions as follows: (Note: commercially purchased certified solutions can be substituted.)

5.3.1. 1.0 N Sulfuric acid: To a 1 liter volumetric flask containing 500 ml distilled water, add 28 ml concentrated sulfuric acid (H_2SO_4), then dilute to the 1 liter mark with distilled water.

5.3.2. 1.0 N Sodium hydroxide: Dissolve 40g sodium hydroxide (NaOH) in a final volume of 1 liter distilled water.

5.3.3. 0.025N Iodine solution:

5.3.3.1. Dissolve 50g KI in 44.5 ml distilled water.

5.3.3.2. Weigh 6.346g I_2 crystals on a watch glass.

5.3.3.3. Hold the watch glass over a 500 ml beaker.

5.3.3.4. Wash the glass with half of the KI solution.

5.3.3.5. Stir to dissolve crystals.

5.3.3.6. Transfer to a 2 liter flask.

5.3.3.7. Wash beaker with remaining KI solution and add distilled water to mark.

5.3.4. 0.025N sodium thiosulfate solution: Dissolve 6.205 g sodium thiosulfate in a final volume 1 liter of distilled water.

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

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

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

5.3.5.3. Discard when starch solution becomes cloudy.

5.4. Measure 100 ml of 1.0N NaOH into the gas bubbler. Attach 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 sodium hydroxide solution. This setting will be used to sample the hydrogen sulfide 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% hydrogen sulfide cylinder.

5.12. Establish the test concentration of 5,000 ppmv or 10,000 ppmv ± 10% hydrogen sulfide by setting the theoretical flow rate of pure hydrogen sulfide 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 hydrogen sulfide concentration has been established by titration and/or dilution /measurement and is stable, testing may begin.

FLOW RATE FOR TEST	FLOW RATE OF PURE HYDROGEN SULFIDE	
LPM	FOR 5000 PPMV	FOR 10000 PPMV
	sccm or mL/min.	
32	160	320
64	320	640

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. Attach the separatory funnel with a rubber stopper to the flask and clamp in place. There must be an air-tight seal between the funnel and flask to prevent the H_2S from escaping.

5.18. Measure 54 ml H_2SO_4 into the separatory funnel.

5.19. Insert the rubber stopper of the aspirator into the top of the separatory funnel.

5.20. Open the stopcock of the separatory funnel and aspirate the H_2SO_4 into the flask.

5.21. Keeping the separatory funnel still in the flask, close the funnel stopcock and remove the aspirator stopper.

5.22. Measure a known volume of 0.025N standard iodine solution (see 5.22.2) into the separatory funnel. The measured amount of iodine added should be in excess of the amount needed.

5.22.1. The hydrogen sulfide cannot be collected directly in iodine because the rate of reaction is too slow to capture it all without loss. The hydrogen sulfide is collected in sodium hydroxide, and released by sulfuric acid at a controlled rate. The sulfuric acid releases hydrogen sulfide when it reacts with the hydrogen sulfide-sodium hydroxide solution in the flask. The hydrogen sulfide thus released is immediately caught in a known excess of iodine. The excess iodine is back titrated with sodium thiosulfate.

5.22.2. For example:

$$\text{Volume } I_2 = \frac{\text{concentration}}{\text{standard factor 305.6 ppmv/ml } I_2}$$

$$\text{Volume } I_2 = \frac{5{,}000 \text{ ppmv}}{305.6 \text{ ppmv/ml } I_2}$$

Volume I_2 = 16.4 ml I_2

5.22.3. Iodine in an amount greater than 16.4 ml should be added, such as 20 ml.

5.23. Record the amount of iodine added.

5.24. Insert the rubber stopper of the aspirator into the top of the separatory funnel.

5.25. Open the stopcock of the separatory funnel and aspirate the iodine into the flask.

5.26. The solution will turn yellow.

5.27. Remove the funnel and stopper.

5.28. Back titrate the excess iodine of the sample concentration with 0.025N sodium thiosulfate dropwise until it is pale yellow.

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

5.30. Continue the titration until the blue color just disappears.

5.31. Obtain the net iodine titration. For example,

net I_2 titration = volume I_2 added - volume $Na_2S_2O_3$ used

net I_2 titration = 20.0 ml - 3.7 ml

net I_2 titration = 16.3 ml I_2

5.32. Calculate the concentration of hydrogen sulfide.

Concentration (ppmv) = net I_2 titration x standard factor

Concentration = 16.3 ml I_2 x 305.6 ppmv/ml I_2

Concentration = 4981 ppmv H_2S

5.33. 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.

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

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

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

5.37. 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.

5.38. If there is another sample to test, repeat steps 5.7 – 5.35.

5.39. 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 G, Section 84.63 (a), (c), (d), and 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 (parts per million volume)[1]	Minimum service life (minutes)
		Gas or vapor	Concentration (parts per million volume)	Flow rate (liters per minute)			
Hydrogen sulfide Front and Back Mounted	As received Equilibrated	H_2S H_2S	10,000 10,000	64 32	3 4	10 10	12 12
Hydrogen sulfide Chin-Style	As received Equilibrated	H_2S H_2S	5,000 5,000	64 32	3 4	10 10	12 12
Hydrogen sulfide Escape	As received Equilibrated	H_2S H_2S	10,000 10,000	64 32	3 4	10 10	12 12

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

7. RECORDS/TEST SHEETS

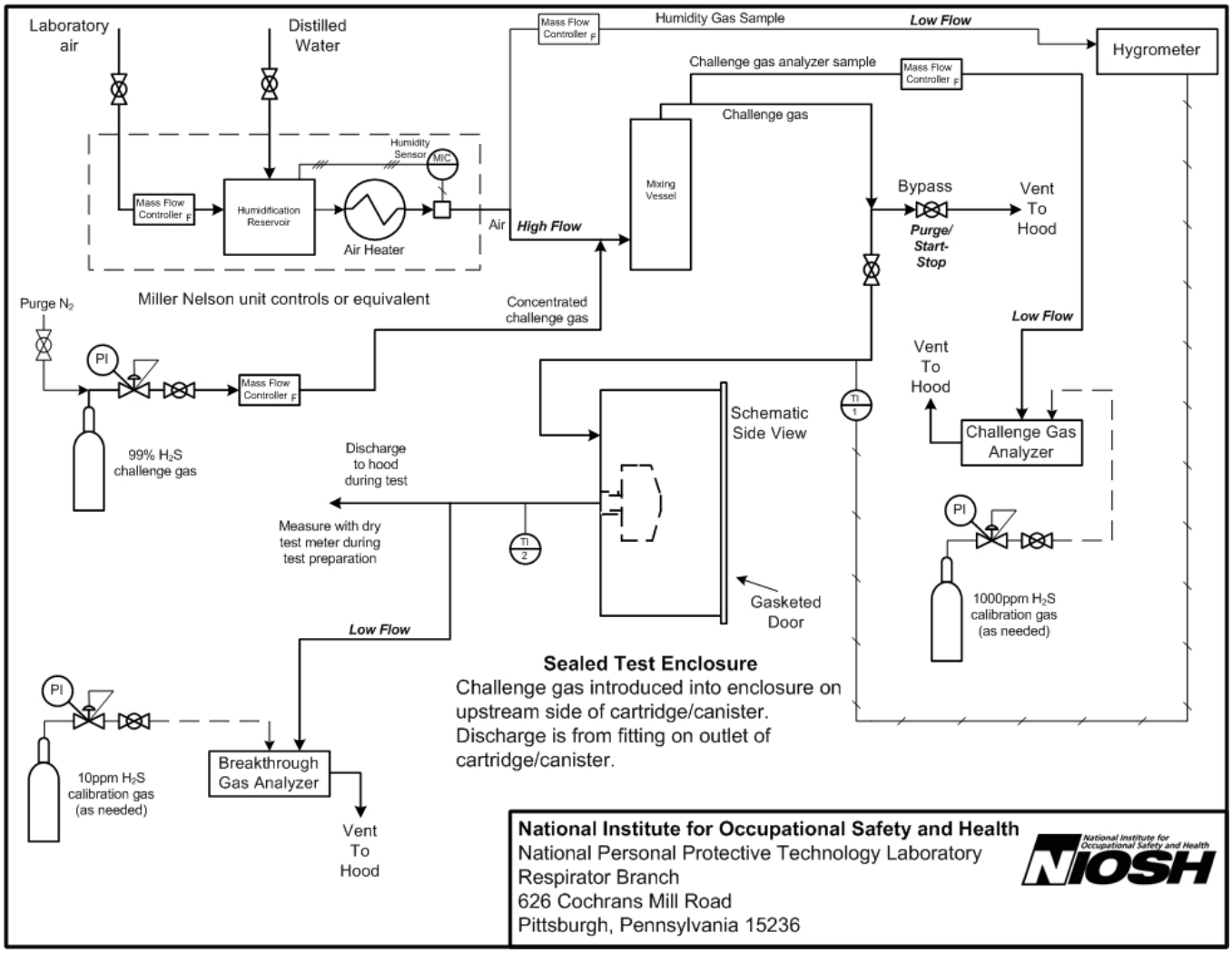
7.1. Record the test data in a format that shall be stored and retrievable.

8. ATTACHMENTS

8.1. Bench Top Set-Up.

8.2. Data Sheet

8.1 Test 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	Test Cond.	Final Time (min)	Leakage (ppmv)	Temperature (°C) Dnstream	Temperature (°C) Upstream	Corrected Time (min)
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:

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	14 March 2008	Significant rewrite of form and clarification of technical content.