



National Institute for Occupational Safety and Health  
National Personal Protective Technology Laboratory  
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Procedure No. TEB-APR-STP-0046A	Revision: 2.0	Date: 19 December 2006
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DETERMINATION OF ORGANIC VAPOR (CARBON TETRACHLORIDE) SERVICE LIFE TEST,  
AIR-PURIFYING RESPIRATORS WITH CARTRIDGES  
STANDARD TESTING PROCEDURE (STP)

1. PURPOSE

This test establishes the procedure for ensuring that the level of protection provided by air-purifying respirators with cartridges submitted for Approval, Extension of Approval, or examined during Certified Product Audits, meet the minimum organic vapor service life test requirements set forth in 42 CFR Part 84, Subpart L, Section 84.207.

2. GENERAL

This STP describes the Determination of Organic Vapor Service Life Test, Air-Purifying Respirators with cartridges 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  $\pm 2\%$  F.S. Temperature control accuracy is  $\pm 1^\circ$  C. Humidity control accuracy is  $\pm 3\%$  R.H.
- 3.1.2. Edge Tech Dew Prime II Hygrometer, Model 2000 or equivalent. Accuracy is  $\pm 0.2^\circ$  C,  $\pm 0.5\%$  RH.
- 3.1.3. Miran Ambient Air Analyzer, Model 1A with closed loop system or equivalent. The analyzer is a single beam variable filter spectrometer with a variable pathlength gas cell.
- 3.1.4. Mass Flow Controllers, Brooks Instruments model series 5850S and 5853S with Read Out and Control Electronics, Brooks Instruments model 0154, variable flow rate depending on use. Accuracy is 0.7% set point & 0.2% FS.
- 3.1.5. American Meter Co. Dry Test Meter Model DTM-325.

Approvals:	<u>1st</u> Level	<u>2nd</u> Level	<u>3rd</u> Level
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- 3.1.6. Amersham Biosciences High Precision Pump, Model P-500 with reservoir. Flow rate range: 1 ml/hr to 400 ml/hr  $\pm$  1.5% of setting.
- 3.1.7. Electronic balance with an accuracy of 0.01grams.
- 3.1.8.. Miller Nelson Research, Model SV-2000 Vaporizer system or equivalent. Used for vaporizing carbon tetrachloride solution.
- 3.1.9. Carbon tetrachloride, ACS grade, 99.9%.
- 3.2. Test fixture for mounting cartridges inside the test chamber. The test fixture used is specific to each manufacturer depending on how the cartridge is mounted to the facepiece. Some cartridges use a standard 40 mm male thread which mates with the test port in the chamber. In some cases the manufacturer is willing to provide a machined adapter for mounting cartridges with unique threads. Alternatively, the cartridge adapters of the respirator are affixed by hot melt glue to a PVC pipe tee of appropriate size which mates with the test port. All adapters are checked for leak-tightness with soap solution.
- 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-1002 – Laboratory Safety Procedures for Carbon Tetrachloride Tests  
TEB-RCT-APR-WI-1102 – Calibration Procedures for Carbon Tetrachloride Tests  
TEB-RCT-APR-WI-1202 – Start-Up and Shut-Down Procedures for Carbon Tetrachloride Tests  
TEB-RCT-APR-WI-1302 – Using the LabView System for Carbon Tetrachloride Tests  
TEB-RCT-APR-WI-1402 – Reporting Results for Carbon Tetrachloride 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	80.54	1.58
EQUIL. 25% RH	186.07	2.89
EQUIL. 85% RH	66.55	8.51

- 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 used in this procedure.

- 4.6 Carbon tetrachloride is a suspected human carcinogen. Containers of carbon tetrachloride are typically used inside the laboratory fume hood. If there is a release of carbon tetrachloride such as a spill outside the hood, sound an alarm, and any personnel in the laboratory should immediately exit from the building. Carbon tetrachloride is considered hazardous waste by EPA and must be disposed of accordingly.

#### 4.7 CARBON TETRACHLORIDE BENCH TEST FOR CARTRIDGES

- 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 84.207.

SAMPLE	CONDITION	EQUILIBRATION CONDITIONS			TEST CONDITIONS			TEST CONCENTRATION	BREAKTHROUGH CONCENTRATION
		FOR 6 HOURS							
		TEMP.	AIRFLOW	R.H.	TEMP.	AIRFLOW	R.H.	PPMV CCl <sub>4</sub>	PPMV CCl <sub>4</sub>
		° C	LPM	%	° C	LPM	%		
1-3	AS RECEIVED	NA	NA	NA	25	64	50	1000	5
4-5	EQUIL. 25% R.H.	25	25	25	25	32	50	1000	5
6-7	EQUIL. 85% R.H.	25	25	85	25	32	50	1000	5

Tolerances:

PARAMETER	TOLERANCE
25°C	± 2.5°C
25 LPM	± 0.25 LPM
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.
1000 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 cartridges 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 For carbon tetrachloride detection with the MIRAN 1A, the wavelength is set to 12.6 µm and the pathlength to 20.25 meters. Minimum detectable concentration is 1.1 ppmv. The closed loop system consists of a stainless steel bellows pump, septum and tubing with a sample volume of 5.64 liters. For calibration procedures, refer to the user manual. Inject 0.11 µL of carbon tetrachloride into closed loop system of the IR as described in the user manual. Refer to section 8.2 for this calculation. Once stabilized, the analyzer should read 5 ppmv. Make adjustment for Labview software to read 5.0 ppmv. Disconnect closed loop system and allow IR to return to zero reading.
- 5.3 Establish the correct humidity and temperature for the sample being tested as per the test requirements in paragraph 4.7.
- 5.4 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 hygrometer flow rates, from the test fixture using the dry test meter.
- 5.5 Weigh the cartridge(s) and record the weight.

- 5.6 Measure initial inhalation and exhalation resistances of the cartridge(s) mounted on the facepiece as described in TEB-APR-STP-003 and TEB-APR-STP-007. Record values on the data sheet.
- 5.7 Make sure diverter valve in the system is diverting the challenge concentration airflow to discharge and not into the testing chamber.
- 5.8 Mount cartridge(s) onto test fixture and place in testing chamber.
- 5.9 Fill pump reservoir with carbon tetrachloride. Insert needle from pump to septum tee in airline.
- 5.9 Set the high precision pump for delivery of calculated carbon tetrachloride to obtain 1000 ppmv at the testing airflow. See appendix 8.2.
- 5.10 Start flow from the high precision pump to the needle.
- 5.11 Record the initial weight of the carbon tetrachloride reserve and start test.
- 5.12 Monitor and record challenge and breakthrough temperatures, challenge RH and breakthrough values and times throughout testing.
- 5.13 Run test until breakthrough of 5.0 ppmv is observed or minimum service life shown in section 6.2 is surpassed by 10%. Weigh and record the final weight of the carbon tetrachloride reservoir.
- 5.14 At end of test, system will automatically direct challenge concentration airflow through diverter valve to discharge.
- 5.15 Calculate and record the challenge concentration of the carbon tetrachloride (see attachment 8.2).
- 5.16 Dismount cartridge(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.
- 5.16 If there is another sample to test, repeat steps 5.5 – 5.15.
- 5.17 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 detector 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 L, Section 84.207.

6.2 Minimum service life requirements for cartridges are shown below.

Cartridge	Test condition	Test atmosphere		Flowrate (l.p.m.)	Number of tests	Penetration <sup>1</sup> (p.p.m.v.)	Minimum life <sup>2</sup> (min.)
		Gas or vapor	Concentration (p.p.m.v.)				
Organic vapors	As received	CCl <sub>4</sub>	1000	64	3	5	25 / 50
Organic vapors	Equilibrated	CCl <sub>4</sub>	1000	32	4	5	25 / 50

<sup>1</sup>Minimum life will be determined at the indicated penetration.

<sup>2</sup>Where a respirator is designed for respiratory protection against more than one type of gas or vapor, as for use in organic vapors and in chlorine, the minimum life shall be 25 minutes. Where a respirator is designed for respiratory protection against one or more than one gas of a single type, as for use in one or two different organic vapors, the minimum life shall be 50 minutes.

7. RECORDS/TEST SHEETS

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

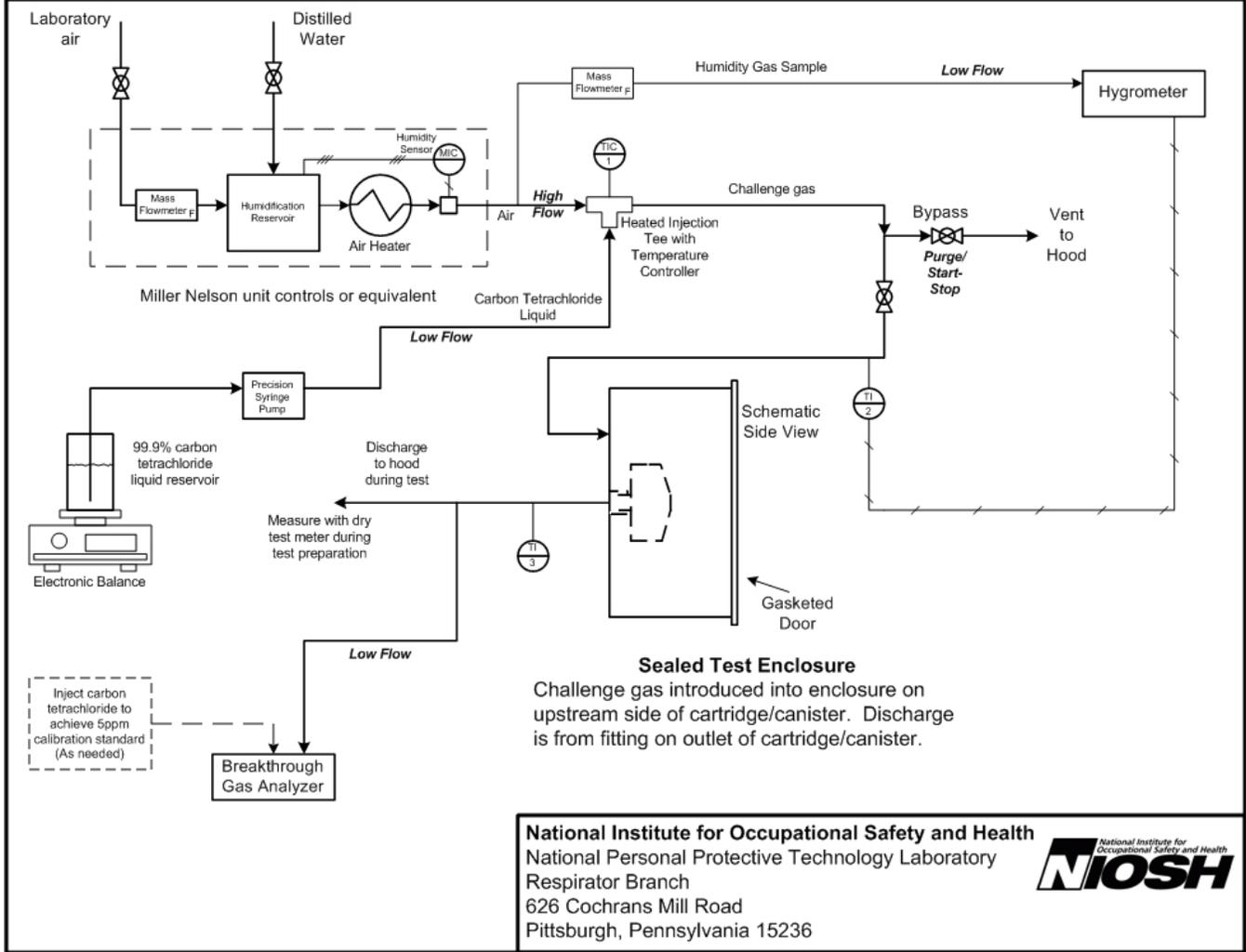
8. ATTACHMENTS

8.1. Bench Top Set-up

8.2. Calculations for carbon tetrachloride

8.3. Data Sheet

8.1 Bench Top Set-Up.



## 8.2: Calculations for carbon tetrachloride

Carbon tetrachloride:

Molecular weight = 153.8 g/mol

1 ppmv = 6.29 mg / m<sup>3</sup> @ 25 °C ; 760 mm Hg

### 1. Calculations for injection rate of carbon tetrachloride.

$$\text{Injection rate (ml/hr)} = \frac{\text{Conc (ppmv)} \times 6.29 \text{ ((mg / m}^3\text{)/ppmv)} \times \text{Airflow (L/min)} \times 60 \text{ min}}{1000 \text{ (mg/g)} \times 1000 \text{ (L/m}^3\text{)} \times 1.59 \text{ (g/ml)}}$$

	64 Lpm	32 Lpm
1000 ppmv	15.19 ml/hr	7.59 ml/hr

### 2. Calculations for actual challenge concentration.

$$\text{Conc (ppmv)} = \frac{\text{amount delivered (g)} \times 24.45 \text{ (L/mol)}}{\text{Airflow (Lpm)} \times \text{Test time (min)} \times 153.8 \text{ (g/mol)}} \times 1,000,000$$

### 3. Calculations for calibrating downstream concentration using closed loop system.

$$\text{Microliter injection amount} = \frac{\text{Conc (ppmv)} \times 5.64\text{L} \times 153.8 \text{ g/mol} \times 1 \times 10^6 \text{ } \mu\text{L} / \text{L}}{1 \times 10^6 \times 24.45 \text{ L/mol} \times 1.59 \text{ g/mL} \times 1000 \text{ mL/L}}$$

For 5 ppmv:

$$\text{Microliter injection amount} = \frac{5\text{ppmv} \times 5.64\text{L} \times 153.8\text{g/mol} \times 1,000,000\text{-}\mu\text{L/L}}{1,000,000 \times 24.45 \text{ L/mol} \times 1.59 \text{ g/mL} \times 1000 \text{ mL/L}}$$

$$= 0.11 \text{ } \mu\text{L}$$

8.3 Data Sheet.

<b>NIOSH</b> <small>National Institute for Occupational Safety and Health</small> <b>RB - RESPIRATOR CERTIFICATION TEAM</b> <b>GAS &amp; VAPOR RESPIRATOR TEST DATA SHEET (Ref.33-48,50,62)</b>									
				STP No.: [ _____ ]					
Task Number: TN- _____			Gas Name: _____						
Manufacturer: _____			Item Tested: _____						

RESISTANCE	Maximum Allowable Resistance (mm of H <sub>2</sub> O)				Actual Resistance (mm of H <sub>2</sub> O)				Result
	Inhalation		Exhalation		Inhalation		Exhalation		
	Test	Initial	Final	Initial	Final	Initial	Final		
1									
2									
3									
4									
5									
6									
7									
Overall Results: Pass _____ Fail _____ Comment: _____									

WEIGHTS AND AIRFLOWS	WEIGHTS (gm)				AIRFLOW (Lpm)			
	Test	Con'd	Conc. (ppmv)	Conc. (ppmv)	Test Rate		(PAPR Only)	
					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)
				Dns tream	Chall enge	
1						
2						
3						
4						
5						
6						
7						

<b>NIOSH</b> <small>National Institute for Occupational Safety and Health</small> Overall Results: Pass _____ Fail _____ Comment: _____ Was all testing equipment in calibration throughout all testing: Yes _____ No _____ Signature: _____ Date: _____									
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 <p><b>NIOSH</b> RB - RESPIRATOR CERTIFICATION TEAM GAS &amp; VAPOR RESPIRATOR TEST DATA SHEET (Ref. 33-48,50,62)</p> <p>Task Number: TN- _____ Gas Name: _____ Manufacturer: _____ Item Tested: _____</p> <p>STP No.: [ _____ ] Page 2</p>
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<p>Additional Comments: Signature: _____ Date: _____</p>
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### Revision History

<b>Revision</b>	<b>Date</b>	<b>Reason for Revision</b>
1.0	March 8, 2002	Historic document
1.1	June 6, 2005	Update header and format to reflect lab move from Morgantown, WV No changes to method
2.0	19 December 2006	Significant rewrite of RCT-APR-STP-0046. Changes affect form and provide clarification of technical content.