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 o-CHLOROBENZYLIDENE MALONONITRILE (CS)
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 o-Chlorobenzylidene malononitrile (CS) service life requirements on gas mask air-purifying respirators submitted for Approval, Extension of Approval, or examined during Certified Product Audits meet the certification requirements set forth in 42 CFR Part 84, Subpart G, Section 84.63(a)(c)(d) and Subpart I, Section 84.110; Volume 60, Number 110, June 8, 1995.

2. GENERAL

This STP describes the Determination of CS Service Life Test, Air-Purifying Respirators 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 or equivalent.

3.1.2. UV Spectrophotometer, Spectronic Model 601 or equivalent.

3.1.3. Supelco Tenax- GC sample tubes custom made to the following specifications: 10cm long, 10 mm outside diameter, 8 mm inside diameter. Front section contains 70 mg 35/60 mesh Tenax-GC; back section contains 35 mg 35/60 Tenax-GC. The Tenax-GC is held in place in the sampling tube by 3 mm silanized glass wool plugs. A 3 mm plug also separates the two sections. The tubes are sealed at both ends.

3.1.4. Digi Sense Scanning Thermocouple Thermometer Model 92800-10.

Approvals:	1st Level	2nd Level	3rd Level

3.1.5. Gelman Acrodisc CR PTFE product number 4226, 1 micron pore size or equivalent.

3.1.6. Supelco 15 ml screw cap vials with hole caps and septa.

3.1.7. "The Gilbrator", Primary Standard Airflow Calibrator, or equivalent.

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

3.1.9. o-Chlorobenzylidene malononitrile (CS).

3.1.10. Hexanes, Spectroanalyzed grade.

3.1.11. Methylene chloride, Spectroanalyzed grade.

3.1.12. 5 ml gas tight glass syringe, volumetric flasks 10-100 ml volume, Eppendorf pippettors or microliter syringes 10 microliter to 500 microliter volume.

3.1.13. Remote control hot plate.

3.1.14. Electronic Balance with an accuracy of 0.001 grams (g).

3.1.15. Teflon tubing ½" diameter for all connections.

3.1.16. Tear gas generator: consists of a steel drum, 5 gallon capacity, approximately 14" in diameter, 18" high, with removable top and ring type closure. Two holes are cut in the lid, one for clean air to enter the chamber and one for the challenge gas to pass through the canister and out into the hood. One ½" hole is cut into the side of the drum near the bottom. The cord from the remote control electric hot plate exits out the hole. Make sure the openings are well sealed with rope caulking, hot melt glue, or other suitable material. A 600 ml beaker containing CS is placed on the hot plate inside the drum and the heat regulated by the remote controller of the hot plate.

3.1.17. Test fixture for mounting cartridges and canisters. The test fixture used is specific to each manufacturer depending on how the cartridge, canister, or powered air-purifying respirator (PAPR) is mounted to the facepiece. The T-end has a 29/42 ground glass joint glued in place. Canisters are tested with their connections glued into the ground glass joint. In most cases the cartridge cups of the respirator are affixed by hot melt glue to a PVC pipe tee of appropriate size. PAPR cartridges and canisters are tested on their blower units or suitable substitution.

3.1.18. 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. CS TEAR GAS BENCH TEST REQUIREMENTS

4.3.1. Resistance to air flow will be measured before each test (see 84.122).

4.3.2. Three "as received" canisters or cartridges will be tested at 64 lpm, continuous air flow, 50 $\pm$ 5 percent relative humidity (RH), approximately 25 degrees Celsius (°C), and 3 ppm (23.1 mg/m^3) of CS.

4.3.3. Two canisters or cartridges 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 $\pm$ 5 percent RH, approximately 25°C, and 64 lpm continuous flow rate containing 3 ppm of CS.

4.3.4. Two canisters or cartridges will be equilibrated at room temperature by passing 85 percent RH air through them at 64 lpm for 6 hours and then testing them at 85 $\pm$ 5 percent RH, approximately 25°C, and 64 lpm continuous flow rate containing 3 ppm of CS.

4.4 CS TEAR GAS BENCH TEST FOR TIGHT FITTING POWERED AIR-PURIFYING RESPIRATORS

4.4.1. Resistance to air flow and PAPR airflow will be measured before each test. (see 84.122).

4.4.2. Three "as received" canisters or cartridges will be tested at 115 lpm, continuous air flow, 50 $\pm$ 5 percent RH, approximately 25 degrees Celsius (°C), and 3 ppm (23 mg/m^3) of CS.

4.4.3. Two canisters or cartridges 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 ± 5 percent RH, approximately 25°C, and 115 lpm continuous flow rate containing 3 ppm of CS.

4.5. CS TEAR GAS BENCH TEST FOR VALVELESS GAS MASKS

4.5.1. Resistance to air flow will be measured before each test (see 84.122).

4.5.2. Three "as received" canisters will be tested at 64 lpm, continuous air flow, 50 ± 5 percent RH, approximately 25 degrees Celsius (°C), and 3 ppm of CS.

4.5.3. Two 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 ± 5 percent RH, approximately 25°C, and 64 lpm continuous flow rate containing 3 ppm of CS.

4.5.4. Two canisters will be equilibrated at room temperature by passing 85 percent RH air through them at 64 lpm for 6 hours and then testing them at 85 ± 5 percent RH, approximately 25°C, and 64 lpm continuous flow rate containing 3 ppm of CS.

4.5.5. One completely assembled gas mask with an as received canister will be tested with a breathing machine with a rate of 24 respirations per minute with a minute volume of 40 liters. A breathing cam with a work rate of 622 Kp-m/min. shall be used. The gas mask will be challenged with an atmosphere at 25°C, 50 ± 5 percent RH, and 3 ppm of CS. The air exhaled through the gas mask will be 35°C ± 2° with 95 ± 3 percent RH.

4.5.6. One completely assembled gas mask with a canister preconditioned at 64 lpm continuous air flow, 25 percent RH, and approximately 25°C, will be tested with a breathing machine with a rate of 24 respirations per minute with a minute volume of 40 liters. A breathing cam with a work rate of 622 Kp-m/min. shall be used. The gas mask will be challenged with an atmosphere at 25°C, 25 ± 5 percent RH, and 3 ppm of CS. The air exhaled through the gas mask will be 35°C ± 2° with 95 ± 3 percent RH.

4.5.7. One completely assembled gas mask with a canister preconditioned at 64 lpm continuous air flow, 85 percent RH, and approximately 25°C, will be tested with a breathing machine with a rate of 24 respirations per minute with a minute volume of 40 liters. A breathing cam with a work rate of 622 Kp-m/min. shall be used. The gas mask will be challenged with an atmosphere at 25°C, 85 ± 5 percent RH, and 3 ppm of CS. The air exhaled through the gas mask will be 35°C ± 2° with 95 ± 3 percent RH.

4.6. **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, calibration, and maintenance of equipment used in this procedure prior to beginning any testing. Malfunctioning equipment must be repaired or replaced and properly set up and calibrated before starting all tests.

5.2. Prepare solutions:

5.2.1. Prepare solvent solution of 20 methylene chloride in hexane. Add 100 ml methylene chloride to a 500 ml volumetric flask. Dilute to mark with hexanes.

5.2.2. CS stock solution 400 ug/ml: accurately weigh approximately 20 mg CS. Record exact weight. Dissolve in 10 ml of 20 methylene chloride in hexane solvent solution. Add to a 50 ml volumetric flask and fill to mark with solvent. Determine exact CS ug/ml stock solution and record.

5.2.3. Example: If 0.0264 g of CS is used for the stock solution.

$$0.0264 \text{ g} = 26400 \text{ ug therefore } \frac{26400 \text{ ug}}{50 \text{ ml}} = 528.0 \text{ ug/ml}$$

528.0 ug/ml = 0.528 ug/ul of CS stock solution

5.3. Prepare standards:

5.3.1. Prepare a group of standards by adding 20, 40, 50, 100, 200, and 400 ul aliquots of stock solution to 10 ml volumetric flasks and dilute to mark with solvent.

5.4. Set up test equipment as shown in Figure 1.

5.5. Turn on UV Spectrophotometer and set wave length to 305 nm and allow to warm up.

5.6. Measure the absorbance of the standards using the 20 percent methylene chloride in hexane solution as the zero absorbance. Check zero absorbance before each concentration sample and reset if strayed.

5.6.1. Example: 20 ul aliquot standard.

20 ul x 0.528 ug/ul CS stock solution = 10.56 ug of total CS in 10 ml or 5.28 ug/5 ml.

5.7. Plot the concentration of each standard as ug/5 ml vs. absorbance.

5.7.1. Example: The absorbance reading of the 20 ul standard is plotted against the standard concentration of 5.28 ug/5 ml.

5.8. Determine desorption coefficient:

5.8.1. Add 75 mg Tenax-GC to three 15 ml screw cap vials. Add 25 ul of stock solution to the resin and let stand overnight. (This result will be ug recovered.)

5.8.2. Add 75 mg Tenax-GC to three 15 ml screw cap vials. Prepare three blanks by adding 25 ul. of solvent to the resin and let stand overnight. (This result will be ug blank)

5.8.3. Next morning add 5 ml solvent to each vial and swirl to thoroughly wet the contents. Let the samples stand for approximately 10 minutes with occasional mixing. Zero the UV spectrophotometer, using the solvent as the zero sample.

5.8.4. Using a 5 ml syringe with an Acrodisc filter attached, withdraw the samples from the vials and put the filtrate in the UV sample cell. Measure and average the absorbance of the three stock vials. Measure and average the absorbance of the three blank vials.

5.8.5. Determine the ug/5 ml reading of the averages from the calibration chart and calculate and record the desorption coefficient as follows:

$$\text{Desorption Coefficient} = \frac{\text{ug recovered - ug blank}}{\text{ug added}}$$

$$\text{Where ug added} = \frac{\text{\% stock solution in ug/ml x 25 ul}}{1000}$$

5.9. Turn on Miller Nelson System and set up CS generation system and let warm up.

5.10. Prior to actual gas testing, a zero blank sample of a canister or cartridge needs to be collected for comparison of breakthrough values. Mount a canister or cartridge onto a test fixture and place in a clean test chamber. Allow clean 64 lpm air to enter the canister/cartridge. Attach the Tenax sample tube to a sample line connected to the Gil Air pump and sample at 1 lpm for 30 minutes in the airstream exiting the canister/ cartridge. Seal both ends after sampling and set aside. This sample will be used as the zero blank.

5.11. Working in a well ventilated hood, cover the bottom of a 600 ml beaker with CS. Cover the top of the beaker with aluminum foil held in place with a rubber band. Punch a ½" hole in the aluminum foil and place the beaker on the hot plate inside the generator. (CS will need to be added at intervals during testing to maintain test concentration.) Seal and lock the lid. Turn on heater.

5.12. Determine test concentration in air: (this must be done at regular intervals during testing to monitor the test concentration.) Mount a concentration canister/cartridge onto the lid.

This will be used to establish the concentration prior to testing of the actual test canister/ cartridge by determining the heat setting that is needed to produce approximately a 3 ppm CS concentration.

5.12.1. Pull a 1liter test concentration sample through the sampling tube. Seal the sample tube until ready to analyze.

5.12.2. Using a small file, score and cut the tube keeping the sections intact. Remove the glass wool plug and discard. Do not add the plugs to the vial. Add the Tenax resin from the front section to a 15 ml vial. Mark vials "front section sample".

5.12.3. Remove the middle plug from the tube and discard. Add the resin from the back section to a different vial. Mark vial "Back Section Sample".

5.12.4. Add 5 ml solvent to both vials and gently swirl to thoroughly wet the contents. Let the samples stand for approximately 10 minutes with occasional swirling.

5.12.5. Withdraw the solution from each vial using separate 5 ml syringes with an attached Acrodisc filter attached for each vial. Then add the filtrate from each vial to a UV cell and measure the absorbance vs. the solvent blank. The solvent blank is redetermined just prior to each absorbance measurement.

5.13. Determine the weight from the calibration chart and calculate the concentration as follows:

$$\text{ug sample} = \text{ug front} + \text{ug back}$$

$$\text{Corrected ug sample} = \frac{\text{ug sample}}{\text{Desorption coefficient}}$$

$$\text{Concentration in mg/m3} = \frac{\text{Corrected ug sample}}{\text{Sample volume in liters}}$$

5.14. Once the test concentration has been established, testing may begin on the actual test, cartridge or canister. Turn off heater and air flow and remove the lid. Remove and discard the concentration canister/cartridge in sealed bag.

5.15. Weigh a new canister/cartridge and record the weight.

5.16. Take inhalation and exhalation resistances of the canister/cartridge mounted on the facepiece at 85 lpm.

5.17. Replace the concentration canister/cartridge with the test canister or cartridge, and lock into place. Turn on air and adjust the heater to the predetermined setting.

5.18. Take a 30 liter sample of the effluent of the canister/cartridge using the sample tubes and Gil-Air pump. Several samples must be taken during testing to determine breakthrough.

5.19. Analyze the sample following steps 5.12.1 through 5.13 using the 30 liter clean air sample from step 5.10 as the zero blank. Replace the zero blank sample to the vial and save for further breakthrough comparisons.

5.20. When the concentration of the effluent of the canister/cartridge reaches 0.05 ppm or the service life exceeds 480 minutes, the test is stopped.

5.21. Turn off hot plate.

5.22. Turn off air.

5.23. Remove test canister/cartridge and place in plastic bag and weigh. Adjust for weight of the plastic bag.

5.24. Turn off 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; 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.

(a) Gas masks including all completely assembled air purifying masks designed for use as respiratory protection during entry into atmospheres not immediately dangerous to life or health or escape only from hazardous atmospheres containing adequate oxygen to support life are described as follows:

(1) Front-mounted or back-mounted gas mask. A gas mask which consists of a full facepiece, a breathing tube, a canister at the front or back, a canister harness, and associated connections.

(2) Chin-style gas mask. A gas mask which consists of a full facepiece, a canister which is usually attached to the facepiece, and associated connections.

(3) Escape gas mask. A gas mask designed for use during escape only from hazardous atmospheres which consists of a facepiece or mouthpiece, a canister, and associated connections.

(b) Gas masks shall be further described according to the types of gases or vapors against which they are designed to provide respiratory protection, as follows:

Type of front-mounted or back-mounted gas mask:

Acid gas[1 2 3]
Ammonia
Carbon monoxide
Organic Vapor [1 2 3]
Other gas(es) and vapor(s) [1 2 3]
Combination of two or more of the above gases and vapors.[1 2 3]
Combination of acid gas, ammonia, carbon monoxide, and organic vapors.[1 2 3]

Type of chin-style gas mask:

Acid gas [1 2 3]
Ammonia
Carbon monoxide
Organic vapor [1 2 3]
Other gas(es) and vapor(s) [1 2 3]
Combination of two or more of the above gases and vapors.[1 2 3]

Type of escape gas mask:

Acid gas [1 2 3 4]
Ammonia [4]
Carbon monoxide
Organic vapor [1 2 3 4]
Other gas(s) and vapor(s) [1 2 3 4]

Combination of two or more of the above gases and vapors.[1 2 3 4]

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[1] Approval may be for acid gases or organic vapors as a class or for specific acid gases or organic vapors.
[2] Not for use against gases or vapors with poor warning properties (except where MSHA or Occupational Safety and Health Administration standards permit such use for a specific gas or vapor), or those which generate high heats or reaction with sorbent materials in the canister.
[3] Use of the gas mask may be limited by factors such as lower explosive limit, toxicological effects,and facepiece fit. Limitations on gas mask service life and sorbent capacity limitations shall be specified by the applicant in instructions for selection, use and maintenance of the gas mask.
[4] Eye protection may be required in certain concentrations of gases and vapors.

(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 Certification and Quality Assurance 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 o-CHLOROBENZYLIDENE MALONONITRILE (CS) 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:

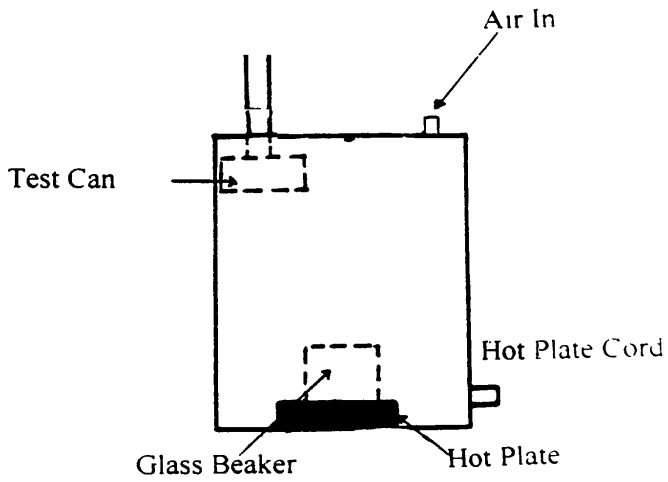
7.3.1. If the failure occurs on a new certification application, or extension of approval application, send a test report to the RCT Leader and prepare the hardware for return to the manufacturer.

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 Tear gas generator.

8.2 Data Sheet.



Tear Gas Generator

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 (°C)**		**Corrected Time (min)**
Test				**Dn stream**	**Upst ream**	
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:

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	19 March 2002	Historic document
1.1	6 June 2005	Update header and format to reflect lab move from Morgantown, WV No changes to method