DETERMINATION OF 2-CHLOROBENZYLIDENE MALONONITRILE SERVICE LIFE TEST, AIR-PURIFYING RESPIRATORS AND POWERED AIR-PURIFYING RESPIRATORS WITH CANISTER(S) STANDARD TESTING PROCEDURE

1. PURPOSE

This document establishes the procedure for ensuring that the level of protection provided by air-purifying respirators (APR) with canister(s) and powered air-purifying respirators (PAPR) with canister(s) submitted for Approval, Extension of Approval, or examined during Certified Product Audits meets the minimum 2- chlorobenzylidene malononitrile (CS) service life test requirements as allowed for in 42 CFR Part 84 Subpart I, Section 84.110(b)(c).

2. GENERAL

This standard testing procedure (STP) describes the named procedure in sufficient detail that a person knowledgeable in the appropriate technical field can select equipment with the necessary resolution, conduct the test, determine whether or not the product passes the test, and create a record of those results, reporting the measurements obtained in a preestablished, standard format.

3. EQUIPMENT/MATERIALS

3.1. Flow-Temperature-Humidity control system: Miller Nelson Research (MNR), model HCS-501-250, or equivalent.

3.2. Chilled mirror hygrometer: Edgetech Instruments, DewMaster, or equivalent. Accuracy is ± 0.2 degrees Celsius (°C) for temperature and dew point.

3.3. UV-Vis Spectrophotometer: Thermo Scientific Genesys 150, or equivalent. Accuracy is 0.5% or 0.002 A, whichever is greater, up to 2.5 A.

3.4. Sample tubes: Supelco ORBO™-402 Tenax® TA, # 20832-U, or equivalent. Made to the following specifications, or equivalent: 100 millimeter (mm) long, 8 mm inside diameter, with two sections of 35-60 mesh Tenax-TA sorbent. Front section contains 100 mg sorbent, and back section contains 50 mg sorbent. The sorbent is held in place in the sampling tube by 3 mm silanized glass wool plugs. The tubes are sealed at both ends.

3.5. Particulate Sampling Filters: GVS Life Sciences, # 1215503, or equivalent. Made to the following specifications: PTFE laminated membrane, 25 mm diameter, 1.0 µm pore size.

3.6. Gas Line Filter Holder: Millipore, # XX4002500, 25 mm filter diameter, or equivalent.

3.7. Mass flow controller: Brooks Instrument, model SLA5850S, or equivalent. With read out and control electronics: Brooks Instrument, model 0254, or equivalent. Accuracy is ± 0.9 percent (%) set point or ± 0.18% full scale, whichever is greater.

3.8. Dry test meter: American Meter Co., Model DTM-325, or equivalent. Accuracy is ± 1% of reading.

3.9. Syringe filters: Millex, #SLFH025, or equivalent. Made to the following specifications: PTFE membrane, 25 mm diameter, 0.45 micrometer pore size.

3.10. Sample vials: Sigma Aldrich, #27210, or equivalent. 7 milliliter (mL) volume, clear glass screw cap vials with hole caps and septa.

3.11. Primary Standard Airflow Calibrator: Sensidyne, Gilian Gilibrator-2, or equivalent.

3.12. Sampling Pump: Sensidyne, Gilian GilAir-3, or equivalent.

3.13. 2-Chlorobenzylidene malononitrile (CS), ≥98% purity.

3.14. Hexanes, ACS spectrophotometric grade, or better.

3.15. Methylene chloride, ACS Certified grade, or better.

3.16. Glassware: 600 mL beaker, 5 mL gas tight glass syringe, volumetric flasks of 10 and 50 mL volume, pipettors or microliter syringes from 10 microliter (µL) to 500 µL volume, and 5 mL bottle dispenser.

3.17. Absorption cuvettes: Hellma, #Z600091, or equivalent. Matched set of 6 cuvettes. Made to the following specifications: 200-2,500 nm spectral range, 3,500 µL volume.

3.18. Hot plate: Generic 4-inch laboratory hot plate.

3.19. Power controller: Glas-Col, model PL312, or equivalent.

3.20. Electronic Balance with a readability of 0.0001 grams (g) and capacity of 150 g, or better, and an Electronic Balance with a readability of 0.01 g and capacity of 1000 g, or better.

3.21. Thermocouple: Type J or better.

3.22. Test chamber: The test chamber consists of an air-tight polycarbonate box approximately 15” W X 18” H X 12” D, with a door opening lined with gasket material, and appropriate inlet, outlet, and sampling port fittings. Additional air-tight penetrations are made into the test chamber for temperature measurement and hot plate power. Upstream temperature is monitored before the inlet of the chamber, box temperature is monitored 1-2” in front of the inlet of the canister within the chamber, and downstream temperature is monitored after the outlet of the chamber. The canister is mounted at the rear of the chamber into a 40 mm female test port with a gasket, using an adapter if necessary. This fixture is not commercially available. The test chamber is shown as Attachment A: Test Chamber Schematic.

3.23. Equilibration test chamber: The equilibration test chamber has interior dimensions of approximately 8 inches (in.) x 21 in. x 21 in. and is constructed of PVC. Within the chamber, there are four evenly spaced canister ports with 40 millimeter (mm) female threads and EPDM rubber gaskets. The chamber front panel is clear acrylic, attaches with

eight ½ in. bolts and wing nuts, and seals air-tight with a rubber gasket. The equilibration airflow enters through a 1-½ in. PVC pipe, mixes in a rear chamber of approximately 3 in. x 21 in. x 21 in. with baffles and enters the main test chamber. The air is pushed through the respirator canister(s) and exits the chamber via a 1-½ in. PVC pipe. Sampling ports are ¼ in. female pipe thread and are located on the inlet and outlet pipes. This fixture is not commercially available.

3.24. Tear gas generator: The hot plate is placed inside the test chamber and controlled with the power controller, which is placed outside of the test chamber. To protect the hot plate, a thin metal pan may be placed on top. A 600mL beaker containing 200-300 mL of CS is placed on the hot plate. A thin aluminum foil sheet with an approximately 1-inch diameter hole may be placed over the beaker to aid in heating the CS. The hot plate should be located as far as possible from the canister inside the test chamber. Tear gas concentration is controlled with the temperature of the hot plate, and the hot plate is manually controlled via the external power controller.

3.25. Test fixture for mounting canister(s): The test fixture used is specific to each manufacturer and respirator model. When possible, respirators using a 40 mm male thread design are directly connected to a 40 mm port in the test chamber. When necessary, a custom test fixture is constructed. The canister interface is removed from the respirator, while keeping the sealing surface and locking interface intact. The interface is affixed by hot melt glue to a PVC pipe fitting of appropriate size, sealed with beeswax, and adapted to a 40 mm male thread fitting with standard pipe fittings. For respirators using a multiple canister configuration, the canisters are tested as a set and a test fixture is constructed using a PVC tee. Respirators with canister(s) and breathing tube shall include the breathing tube as part of the test fixture. When possible, PAPR systems with canister(s) shall use the respirator body and breathing tube as the test fixture, with a 40 mm male thread adapter affixed to the breathing tube by hot melt glue and sealed with beeswax.

4. TESTING REQUIREMENTS AND CONDITIONS

4.1. Equipment shall be operated and calibrated in accordance with the testing laboratory's operation and calibration procedure(s) or the manufacturer's operation and maintenance manual(s). All measuring equipment utilized for this testing should have been calibrated using a method traceable to the National Institute of Standards and Technology (NIST), when available.

4.2. Normal laboratory safety practices must be observed. Refer to Safety Data Sheets (SDS) for the proper protection and care in handling, storing, and disposing of the chemicals used in this procedure. All applicable local, state, and federal regulations for the safe handling and use of hazardous substances shall be followed.

4.3. 2-chlorobenzylidene malononitrile is commonly known as CS tear gas and is a potent eye and skin irritant. Containers of CS are typically used inside the laboratory fume hood. Care should be taken to avoid all skin contact, and an appropriate method of bodily cleaning and remediation should be readily available.

4.4. 2- CHLOROBENZYLIDENE MALONONITRILE BENCH TEST FOR CANISTER(S)

4.4.1. For any APR with canister(s):

For the complete APR, exhalation resistance to airflow will be measured before each test, and inhalation resistance to airflow will be measured before and after each test. The STPs are described in TEB-APR-STP-0003 and TEB-APR-STP-0007.

4.4.2. For any PAPR with canister(s):

For the complete tight-fitting PAPR, exhalation resistance to airflow will be measured before each test, and airflow and inhalation resistance to airflow will be measured before and after each test. The STPs are described in TEB-APR-STP-0003, TEB-APR-STP-0007, and TEB-APR-STP-0012.

4.4.3. For respirators designed for a multiple canister configuration, the canisters are tested as a set. Each set will be tested together on a single test fixture and will be treated as a single sample.

4.4.4. Test conditions as allowed for by 42 CFR 84.110, 84.126, and 84.175(b) are described in Tables 1 through 3.

4.4.4.1. The breakthrough concentration is the maximum allowable penetration for the respirator.

4.4.4.2. A respirator canister For One Type provides respiratory protection against CS and may also include protections for only other acid gases or vapors.

4.4.4.3. A respirator canister For More Than One Type provides respiratory protection against CS, may also include protections for other acid gases or vapors, and must also include protection for at least one other gas or vapor of another type.

Table 1: Test Conditions and Minimum Service Life Criteria for Air-Purifying Respirator with Canister(s)

Number of Samples	**Condition**	**Equilibration Conditions** For 6 hours			**Test Conditions**				**Concentration**		**Minimum Service Life**
		Temp.	**Airflow**	**RH**	**Upstream Temp.**	**Box Temp.**	**Airflow**	**RH**	**Challenge**	**Breakthrough**	**For One Type or More Than One Type**
		°C	**LPM**	**%**	**°C**	**°C**	**LPM**	**%**	**ppm**	**ppm**	**min**
3	As-received	NA	NA	NA	25	42	64	50	3	0.05	480
2	Equilibrated 25 %RH	25	64	25	25	42	64	25	3	0.05	480
2	Equilibrated 85 %RH	25	64	85	25	42	64	85	3	0.05	480

Table 2: Test Conditions and Minimum Service Life Criteria for Tight-Fitting Powered Air-Purifying Respirator with Canisters(s)

Number of Samples	Condition	Equilibration Conditions For 6 hours			Test Conditions				Concentration		Minimum Service Life
		Temp.	Airflow	RH	Upstream Temp.	Box Temp.	Airflow	RH	Challenge	Breakthrough	For One Type or More Than One Type
		°C	LPM	%	°C	°C	LPM	%	ppm	ppm	min
3	As-received	NA	NA	NA	25	42	115	50	3	0.05	480
2	Equilibrated 25 %RH	25	115	25	25	42	115	25	3	0.05	480
2	Equilibrated 85 %RH	25	115	85	25	42	115	85	3	0.05	480

Table 3: Tolerances for Test Conditions

Parameter	Tolerance
25 °C[1]	± 2.5 °C
42 °C	± 8 °C
64 LPM[2]	± 1.0 LPM
115 LPM[2]	± 1.0 LPM
25 %RH[3]	± 5 %RH
50 %RH[3]	± 5 %RH
85 %RH[3,4]	+0/-5 %RH
3 ppm	± 10%

[1] Temperature specification is for upstream temperature only. There is no requirement or specification for downstream temperature.
[2] Tolerance for airflow rates exceeds specification of the MNR because flow rates are calibrated for every test. This improves the precision of the measurement and allows for tighter tolerance on short-term drift.
[3] RH is determined using the upstream temperature value.
[4] RH levels greater than 85% are difficult to maintain and may cause rapid degradation of service life.

4.4.5. All as-received canisters shall be stored in containers same as or similar to the original packaging prior to testing.

4.4.6. All equilibrated canisters shall be stored in sealed containers, kept in a position such that the direction of airflow would be horizontal, and at room temperature, and testing shall begin within 18 hours after equilibration.

5. PROCEDURE

5.1. Set up the test equipment as shown in Attachment B: Test Schematic.

5.2. Prepare solvent solution.

5.2.1. Prepare a solvent solution consisting of 20% methylene chloride in hexane, by volume. For example, combine 100 mL methylene chloride and 400 mL hexane in a dispenser bottle. Mix gently to combine solution.

5.3. Characterize the Tenax TA sample tubes to determine the sorbent desorption coefficient prior to testing.

NOTE: Characterization is only required once per each lot of sample tubes.

5.3.1. Prepare CS stock solution of approximately 400 µg/mL in solvent solution.

5.3.1.1. Precisely weigh approximately 20 milligrams (mg) of CS. Dissolve in a small amount of solvent solution and transfer to a 50 mL volumetric flask. Dilute solution to 50 mL fill mark with solvent solution. Determine exact CS µg/µL stock solution and record.

5.3.1.2. Example: If 0.0264 g of CS is used to make the stock solution:

$$0.0264\ \text{g} = 26400\ \mu\text{g}$$

$$\frac{26400\ \mu\text{g}}{50\ \text{mL}} = 528.0\ \mu\text{g/mL}$$

$528.0\ \mu\text{g/mL} = 0.528\ \mu\text{g/µL}$ CS stock solution

5.3.2. Prepare standard solutions of CS in solvent solution.

5.3.2.1. Prepare a set of standard solutions by adding precisely 20, 40, 50, 100, 200, and 400 µL aliquots of stock solution to each of six 10 mL volumetric flasks. Dilute solutions to 10 mL fill mark with solvent solution solvent. Determine the exact µg/5 mL for each standard solution and record.

5.3.2.2. Example: For 20 µL aliquot standard solution:

$20\ \mu\text{L} \times 0.528\ \mu\text{g/µL}$ CS stock solution $= 10.56\ \mu\text{g}$ CS in 10 mL

$10.56\ \mu\text{g}$ CS in 10 mL $= 5.28\ \mu\text{g}$ CS in 5 mL

5.3.3. Generate a standard curve with a best-fit line and equation.

5.3.3.1. Turn on UV-Vis spectrophotometer, set wavelength to 305 nanometers (nm), and allow detector to equilibrate per manufacturer's instructions.

5.3.3.2. Measure the absorbance of each of the six standard solutions of CS in solvent solution.

5.3.3.2.1. A clean cuvette filled with only solvent solution serves as the zero blank. The UV-Vis spectrophotometer will automatically measure and deduct the zero blank absorbance value from each measurement.

5.3.3.2.2. A portion of each standard solution is added to a clean cuvette and the absorbance is measured by the UV-Vis spectrophotometer set to a wavelength of 305 nm.

5.3.3.3. Using typical graphing software, generate a graph with the µg/5 mL of each standard solution on the x-axis and the measured absorbance for each standard solution on the y-axis.

NOTE: The "µg/5 mL" unit is used because all subsequent samples in this procedure require the determination of µg CS diluted in a 5 mL sample.

5.3.3.4. Use the software to generate a best-fit line of the data, and determine the equation of the line in the standard format of y=mx+b, where y is the absorbance, m is the slope of the line, x is the µg/5 mL, and b is the value at which the line intersects the y-axis.

5.3.4. Determine the desorption coefficient of the Tenax TA sorbent.

5.3.4.1. Remove Tenax TA sorbent from new sample tubes, and add 75 ± 1 mg to each of six sample vials. Ensure that there are no contaminants or glass wool present in the vial.

5.3.4.2. For three sample vials, add 25 µL of solvent solution to the sorbent, ensuring that the liquid is delivered to the sorbent and does not contact the glass vial. These will serve as the desorption blank samples.

5.3.4.3. For three sample vials, add 25 µL of CS stock solution to the sorbent, ensuring that the liquid is delivered to the sorbent and does not contact the glass vial. These will serve as the recovered CS samples.

5.3.4.4. Allow the six sample vials to sit undisturbed for a minimum of 12 hours.

5.3.4.5. Add 5 mL of solvent solution to each of the six sample vials to desorb the samples. Let the samples stand for approximately 10 minutes, with occasional swirling/mixing to thoroughly wet the contents. Desorption is considered complete and stable after 10 minutes have elapsed.

5.3.4.6. Using a 5 mL glass syringe with a syringe filter attached, withdraw a portion of the liquid from each sample vial and place the filtrate into individual cuvettes.

5.3.4.7. Measure the absorbance of each of the six samples in cuvettes.

5.3.4.7.1. A clean cuvette filled with only solvent solution serves as the zero blank. The UV-Vis spectrophotometer will automatically measure and deduct the zero blank absorbance value from each measurement.

5.3.4.7.2. The absorbance is measured by the UV-Vis spectrophotometer set to a wavelength of 305 nm.

5.3.4.8. Determine the average absorbance values for the three desorption blank samples and the three recovered CS samples.

5.3.4.9. Using the standard curve equation derived in 5.3.3.4 above, calculate the µg/5 mL value for the desorption blank and recovered CS samples.

5.3.4.9.1. Example: If the equation derived from the standard curve is y=0.0158x-0.0176 and 0.053 is the measured absorbance value:

Where:

y is the absorbance value and = 0.053
0.0158 is the example slope of the standard curve
x is the µg/5 mL value to be calculated
-0.0176 is the example y-intercept of the standard curve

$$x = \frac{0.053 + 0.0176}{0.0158} = 4.468\ \mu g/5\ mL$$

5.3.4.10. Calculate and record the desorption coefficient using the following equation:

$$\text{Desorption Coefficient} = \frac{\mu\text{g/5 mL recovered CS} - \mu\text{g/5 mL blank}}{\mu\text{g added}}$$

Where:

$$\mu\text{g added} = \mu\text{g/}\mu\text{L stock solution} \times 25\ \mu\text{L stock solution added}$$

5.4. Determine a test system zero blank value.

5.4.1. Prior to canister testing, a zero blank value of the test system shall be determined. This step will negate any potential measured breakthrough CS concentration due to contamination of the test system.

5.4.2. Mount a new, clean canister(s) onto the test fixture in the test chamber.

5.4.3. Establish the testing conditions as described in section 5.7 below.

5.4.4. Connect the sample tube to the test system.

5.4.4.1. Attach a sample tube to the downstream sampling port on the exhaust line from the canister(s). Rope caulk or a similar putty-like material is typically sufficient to obtain an adequate connection of the sample tube to the sampling port.

5.4.4.2. Connect a sample line from the sample tube to the sampling pump.

5.4.5. Set the sampling pump to a flow rate of 1.0 LPM and pull air through the sample tube for 30:00 minutes.

5.4.6. Immediately after sample collection, disconnect the sample tube and seal both ends until ready to analyze.

5.4.7. Refer to section 5.18 for sample tube analysis.

5.5. For equilibration of samples prior to testing against CS, follow sections 5.7 through 5.11, 5.14 through 5.16, 5.20, and 5.21 below. Repeat as necessary for additional samples.

5.5.1. Equilibration of samples is completed in a separate and clean equilibration test chamber, which has not been used for CS testing. The CS test chamber is not to be used for equilibration or samples.

5.6. For testing against CS, follow sections 5.7 through 5.19, and 5.22 through 5.24 below. Repeat as necessary for additional samples.

5.7. Establish the testing or equilibration conditions to the required values described in section 4.4 above.

5.7.1. Airflow is controlled and adjusted using the MNR, and measured at the test chamber using the dry test meter prior to each test. Use caution as to account for any flow losses from the hygrometer or sampling pump, and do not expose the dry test meter to CS.

5.7.2. Relative humidity is controlled and adjusted using the MNR, and continuously measured using the chilled mirror hygrometer. A stream of clean air is fed to the hygrometer from near the outlet of the MNR, and is controlled to approximately 0.8 LPM using a mass flow controller. The temperature probe is mounted in the inlet pipe of the test chamber.

5.7.3. Temperature is typically determined by the laboratory environmental conditions. It is possible to control and adjust heating of the test system using the MNR, but may result in condensation within the test system if the laboratory temperature is not adequate. Upstream temperature is continuously measured using the chilled mirror hygrometer, with the temperature probe mounted in the inlet pipe of the test chamber. Downstream temperature is continuously measured using a thermocouple probe mounted in the outlet pipe of the test chamber. NOTE: Temperature differentials in and around the test system may cause condensation and inaccurate test conditions.

5.8. Weight of the canister(s) is measured in the initial as-received state, following equilibration, and following test completion.

5.9. Measure airflow and resistance to airflow of the respirator before and after each test as described in section 4.4 above. The measurements before each test are taken prior to equilibration.

5.10. Ensure that the airflow has been diverted and is not entering the test chamber.

5.11. Mount canister(s) onto an appropriate test fixture and mount in test chamber.

5.12. Establish the challenge concentration to the required value described in section 4.4 above.

5.12.1. 200-300 mL of solid CS is placed in a beaker on the hot plate inside the test chamber. The CS is heated, which liquifies the CS and generates CS vapor in the test system.

5.12.1.1. A thin metal pan may be placed on the hot plate to protect it from an accidental spillage of CS.

5.12.1.2. A thin aluminum foil sheet with an approximately 1-inch diameter hole may be placed over the CS to aid in heating the CS.

5.12.2. Challenge concentration is controlled by the temperature of the hot plate inside the test chamber, and the hot plate is manually controlled by the external power controller.

5.12.2.1. The required temperature and temperature setting to reach the desired challenge concentration are system dependent. Values are approximately reproduceable, and manual adjustments should be expected.

5.12.3. Seal the test chamber and allow the test system to equilibrate.

5.13. Determine the challenge concentration of CS in the test system.

5.13.1. Place a particulate sampling filter in the gas line filter holder and connect it to the upstream sampling port on the test chamber. Use an appropriate tube fitting for the connection, typically a Swagelok® port connector. Ensure that the sampling filter and filter holder are installed in the correct orientation, per the manufacturers' instructions.

5.13.2. Connect the sample tube to the outlet of the filter holder.

5.13.2.1. Attach a sample tube to the filter holder connected to the test chamber. A short length of flexible laboratory tubing is typically sufficient to obtain an adequate connection of the sample tube to the sampling port.

5.13.2.2. Connect a sample line from the sample tube to the sampling pump.

5.13.3. Set the sampling pump to a flow rate of 1.0 LPM and pull air through the sampling filter and sample tube for 1:00 minute.

5.13.4. Immediately after sample collection, disconnect the filter holder and sample tube from the test system. Seal both ends of both the filter holder and sample tube until ready to analyze.

5.13.5. The challenge concentration shall be periodically measured throughout the test.

5.13.5.1. A minimum of three challenge concentration measurements are required, capturing the beginning of the test, near the middle of the test, and the end of the test.

5.14. Testing or equilibration may begin once all parameters have stabilized at the required values.

5.15. Testing or equilibration begins when the airflow is directed into the test chamber.

5.16. Periodically monitor and record the challenge concentration and breakthrough concentration, and continuously monitor the RH, upstream temperature, downstream temperature, and box temperature throughout testing or equilibration. Make adjustments as necessary to maintain the required conditions.

5.17. Determine the breakthrough concentration of CS through the canister(s).

5.17.1. Connect the sample tube to the test system.

5.17.1.1. Attach a sample tube to the downstream sampling port on the exhaust line from the canister(s). Rope caulk or a similar putty-like material is typically sufficient to obtain an adequate connection of the sample tube to the sampling port.

5.17.1.2. Connect a sample line from the sample tube to the sampling pump.

5.17.2. Set the sampling pump to a flow rate of 1.0 LPM and pull air through the sample tube for 30:00 minutes.

5.17.3. Immediately after sample collection, disconnect the sample tube and seal both ends until ready to analyze.

5.17.4. The breakthrough concentration shall be periodically measured throughout the test.

5.17.4.1. At minimum, samples shall be taken at the beginning and end of the test, spanning the full 8-hour test time criteria, or until the breakthrough criteria is exceeded.

5.18. Sample tube and particulate sampling filter analysis:

5.18.1. Using a small file or glass cutter, score and snap the sample tube above the front section of Tenax TA sorbent, taking caution to not shatter the tube. Using a dental pick (or similar), remove the glass wool plug and discard. Do not add the plug to the vial. Add the sorbent from the front section of the sampling tube to a vial. Mark the vial as the front section sample.

5.18.2. Remove the middle glass wool plug from the sample tube and discard. Add the sorbent from the back section of the sample tube to a different vial. Mark the vial as the back section sample.

5.18.3. Using tweezers (or similar), remove the sampling filter from the filter holder, taking car to not dislodge any collected CS on the filter. Add the sampling filter to a vial. Mark the vial as the filter sample.

5.18.4. Add 5 mL of solvent solution to each of vial to desorb the samples. Let the samples desorb for approximately 10 minutes, with occasional swirling/mixing to thoroughly wet the contents. Ensure that the sampling filter is fully wetted and submerged in the solvent solution. Desorption is considered complete and stable after 10 minutes have elapsed.

5.18.5. Using a 5 mL glass syringe with a syringe filter attached, withdraw a portion of the liquid from each sample vial and place the filtrate into individual cuvettes. Each vial requires a separate syringe and syringe filter to avoid contamination.

5.18.6. Measure the absorbance of the filter sample, front section sample, and back section sample in cuvettes.

5.18.6.1. A clean cuvette filled with only solvent solution serves as the zero blank. The UV-Vis spectrophotometer will automatically measure and deduct the zero blank absorbance value from each measurement.

5.18.6.2. The absorbance is measured by the UV-Vis spectrophotometer set to a wavelength of 305 nm.

5.18.7. Using the standard curve equation derived in 5.3.3.4 above, calculate the µg/5 mL value for the sample tube. The µg/5 mL value is equivalent to the uncorrected µg CS value collected for the sample.

5.18.7.1. See section 5.3.4.9.1 for example calculation.

5.18.7.2. For each sample tube, the measured concentration is the sum of the filter sample, front section sample, and back section sample.

NOTE: If the concentration of the back section sample exceeds 25% of the concentration of the front section sample within a single sample tube, the probability of sample loss exists and the sample should be discarded and repeated.

5.18.8. Apply the desorption coefficient to determine the corrected µg CS value collected for the sample using the following equation:

$$\text{Corrected µg CS} = \frac{\text{µg CS collected}}{\text{Desorption Coefficient}}$$

5.18.9. Convert the µg CS value to ppm using the following equation:

$$\text{ppm} = \frac{\text{µg CS} \times 24.45\ \text{L/mol}}{\text{L} \times 188.6\ \text{g/mol}}$$

Where:

L is the volume of air sampled from the test chamber. L = flow rate × time
24.45 L/mol is the volume of 1 mole of gas at 1 atmosphere and 25 °C
188.6 g/mol is the molecular weight of 2-chlorobenzylidene malononitrile

5.19. For the breakthrough concentration, subtract the test system zero blank calculated in section 5.4 above.

NOTE: This is done for the breakthrough concentration only, and not for the challenge concentration.

5.19.1. Example: If the test system zero blank is calculated to be 0.005 ppm and the breakthrough concentration sample tube is calculated to be 0.052 ppm:

$$0.052 \text{ ppm} - 0.005 \text{ ppm} = 0.047 \text{ ppm}$$

The initial measured breakthrough concentration of 0.052 ppm appears to be a failing result, but the corrected actual breakthrough concentration is 0.047 ppm and is a passing result.

5.20. Equilibration is complete when six hours has elapsed.

5.21. When equilibration is complete, divert airflow away from the equilibration test chamber. Remove the canister(s) from the test chamber. Measure the equilibrated weight as described above. The equilibrated sample is now ready to be used as a test sample.

5.22. A test is complete when the breakthrough concentration limit has been exceeded or the minimum service life has elapsed, whichever comes first.

5.23. When testing is complete, divert airflow away from the test chamber and turn off the external power controller. Remove the canister(s) from the test chamber. Measure the final weight, airflow, and resistance to airflow as described above.

5.24. At the completion of all testing or at the end of a work shift, allow the CS to cool to room temperature and solidify. Remove the test fixture from the test chamber, and plug the test port or install a clean, blank canister.

5.24.1. CS can typically be reused for several tests, at the technician's discretion.

6. PASS/FAIL CRITERIA

6.1. The 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 are shown in Tables 1 and 2 above.

6.3. A failure occurs when the measured test breakthrough concentration exceeds the stated breakthrough concentration limit prior to the test reaching the stated minimum service life time.

6.4. A failure occurs when the airflow or resistance to airflow before or after a test fails to meet the requirements set forth in 42 CFR Part 84 Subpart I, Section 84.122, Subpart K, 84.175(b), and Subpart L, 84.203, and described in TEB-APR-STP-0003, TEB-APR-STP-0007, and TEB-APR-STP-0012.

7. RECORD/TEST SHEETS

7.1. Record the test data in a format that shall be stored and retrievable. Data should be reported similar to Attachment C: Sample Test Data Sheet for 2-Chlorobenzylidene Malononitrile Service Life Test.

8. LIST OF ABBREVIATIONS AND ACRONYMS

Table 6: List of Abbreviations and Acronyms Used Within This Document

Abbreviation or Acronym	Definition
°C	degrees Celsius
%	percent
APR	air-purifying respirator
CFR	Code of Federal Regulations
CS	2-chlorobenzylidene malononitrile, CS tear gas
g	gram(s)
LPM	liters per minute
min	minute(s)
mg	milligram(s)
mL	milliliter(s)
mL/min	milliliters per minute
mm	millimeter(s)
MNR	Miller Nelson Research Flow-Temperature-Humidity control system
mol	mole
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standard and Technology
Nm	nanometer(s)
NPPTL	National Personal Protective Technology Laboratory
PAPR	powered air-purifying respirator
ppm	parts per million
RH	relative humidity
SDS	safety data sheet
STP	standard testing procedure
µg	microgram(s)
µL	microliter(s)

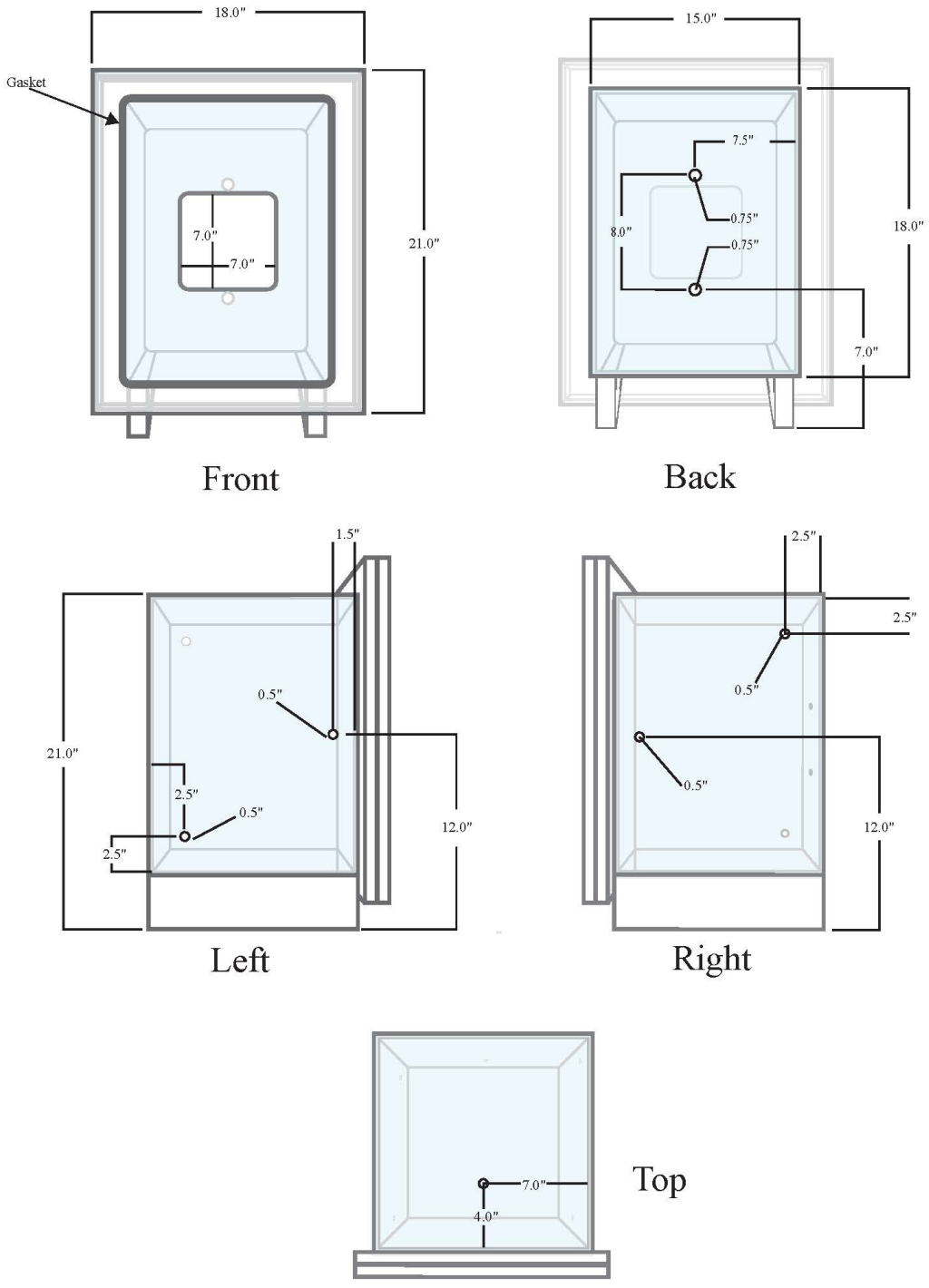
9. ATTACHMENTS

9.1. Attachment A: Test Chamber Schematic

9.2. Attachment B: Test Schematic

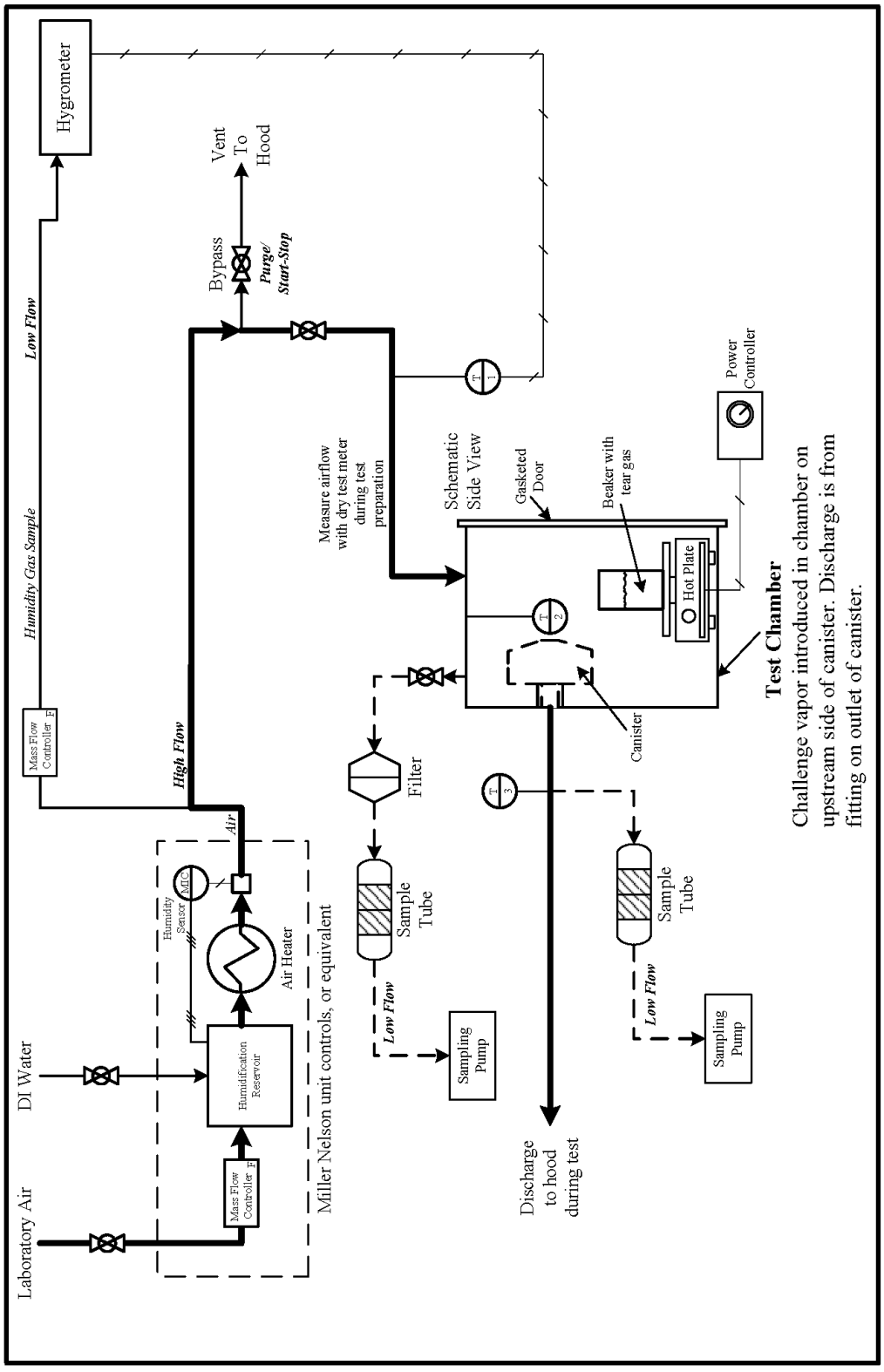
9.3. Attachment C: Sample Test Data Sheet for 2-Chlorobenzylidene Malononitrile Service Life Test

Attachment A: Test Chamber Schematic



Internal Dimensions: 14.0" width, 17.0" height, 12.0" depth • Wall thickness 0.5"

Attachment B: Test Schematic



Attachment C: Sample Test Data Sheet for 2-Chlorobenzylidene Malononitrile Service Life Test

National Institute for Occupational Safety and Health

Test Data Sheet

Task Number:
Test:
Manufacturer:
Item Tested:

STP No.:

RESISTANCE

Respirator Type:

Test	Maximum Allowable Resistance (MM of H2O) Inhalation Initial	Inhalation Final	Exhalation Initial	Exhalation Final	Actual Resistance (MM of H2O) Inhalation Initial	Inhalation Final	Exhalation Initial	Exhalation Final	Result
1									
2									
3									
4									
5									
6									
7									

Overall Result:

Test	WEIGHTS (gm.) As Received	Pre-Conditioned	Water Gain	Final Weight	Concentration (ppm)	AIR FLOW (Lpm) RH%	Minimum Allowable	Initial	Final	Result
1										
2										
3										
4										
5										
6										
7										

Overall Result:

DATA TABLE

Test	Test Condition	Breakthrough (ppm)	Temperature (C) Dnstream	Upstream	Minimum Service Life (min.)	Final Time (min.)	Result
1	As Received						
2	As Received						
3	As Received						
4	25% Preconditioned						
5	25% Preconditioned						
6	85% Preconditioned						
7	85% Preconditioned						

Overall Result: PASS

Signature:

Date: _____

Test Administrator

Form revision: 1.1 February 8, 2023

Task Number:
Test:
Manufacturer:
Item Tested:

STP No.:

Comments:

Was all equipment verified to be in calibration throughout all testing?	◯ Yes	◯ No

Signature:

Date: _____

Test Administrator

Form revision: 1.1 February 8, 2023

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
2.0	23 July 2026	Significant editorial rewrite to update language and make clarifications to procedure. Updated document has been renumbered as NPPTL-APR-STP-0050. Changes affect document format and header. Updates to the procedure to specify temperature range and modify test schematic to match current lab practices. Clarification of tolerances for all measured values.