National Institute for Occupational Safety and Health National Personal Protective Technology Laboratory P.O. Box 18070 Pittsburgh, PA 15236		
Procedure No. RCT-APR-STP-0039	Revision: 1.1	Date: 28 June 2005

DETERMINATION OF FORMALDEHYDE 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 formaldehyde service life requirements on chemical cartridges and gas masks air-purifying respirators submitted for Approval, Extension of Approval, or examined during Certified Product Audits meet the minimum certification standards set forth in 42 CFR Part 84, Subpart G, Section 84.63(a)(c)(d), Subpart I, Section 84.110(c), and Subpart L, Section 84.190(b); Volume 60, Number 110, June 8, 1995.

2. GENERAL

This STP describes the Determination of Formaldehyde 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. Interscan Corporation Model 1168 Formaldehyde Analyzer or equivalent.

3.1.3. VICI Metronics Dynacalibrator Permeation system, Model 340-35-X, with 1 ppm HCHO certified gas permeation tube.

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

3.1.5. Sage syringe pump, Model 355 or equivalent.

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

3.1.7. Graduated impinger, 25ml.

Approvals:	1st Level	2nd Level	3rd Level

3.1.8. Erlenmeyer flasks, 250 to 500 milliliters (ml), and 600 ml beaker.

3.1.9. Pipets, 5 and 10 ml.

3.1.10. Hamilton Gas Tight Syringes, 50ml- Hamilton syringe with T-Valve Model 86727 and Hamilton Teflon Tubing Assembly 90628.

3.1.11. Three necked round bottom flask (250 ml) with heating mantle and temperature controller.

3.1.12. Heated vaporizing element to suspend in the three necked round bottom flask. The formaldehyde solution is delivered via the syringe and tubing assembly to the element, where it is vaporized into the air-stream. This is not commercially available.

3.1.13. Electronic balance with accuracy of 0.001 grams (g).

3.1.14. Vaisala model HMI 31 humidity indicator.

3.1.15 Para-formaldehyde, purified grade.

3.1.16. Sodium Thiosulfate (granular) or 0.1N certified sodium thiosulfate solution.

3.1.17 Starch, Soluble Potato, Powder.

3.1.18. Iodine, 0.1N certified solution.

3.1.19. Sodium Bisulfite, granular.

3.1.20. Sodium Carbonate, anhydrous powder.

3.1.21. Glacial acetic acid.

3.1.22. Heated magnetic stirrer and stir bar.

3.2. 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), mouth bit, etc. 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 if possible, or on suitable substitutions, if the unit is too large for the test chamber.

3.3. The test chamber consisting of a 12" x 11½" x 7" air tight box, made of ½" plexiglass with 2 hinge type locks on the door opening lined with gasket material. A ½" hole is located on the backside of the test chamber for the introduction of the test concentration

and a 1½" hole on the top for the exit of the test fixture and to detect the breakthrough concentration. This fixture is not commercially available.

3.4. 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. FORMALDEHYDE BENCH TEST FOR CARTRIDGES

4.3.1. Resistance to air flow will be taken before and after each test (see 84.203).

4.3.2. Three "as received" cartridges (or pairs of cartridges) will be tested at 64 lpm, continuous air flow, 50 $\pm$ 5 percent relative humidity (RH), approximately 25 degrees Celsius (^{O}C), and 100 ppm HCHO.

4.3.3. Two cartridges or pairs of cartridges will be equilibrated at room temperature by passing 25 percent RH air through them at 25 lpm for 6 hours and then testing them at 25 percent RH, approximately 25^{O}C, and 64 lpm continuous air flow containing and 100 ppm HCHO.

4.3.4. Two cartridges or pairs of cartridges will be equilibrated at room temperature by passing 85 percent RH air through them at 25 lpm for 6 hours and then testing them at 85 percent RH, approximately 25^{o}C, and 64 lpm continuous air flow containing and 100 ppm HCHO.

4.4. FORMALDEHYDE BENCH TEST FOR CANISTERS

4.4.1. Three "as received" canisters will be tested at 64 lpm, continuous air flow,

50 ± 5 percent RH, approximately 25°C, containing 500 ppm HCHO for chin style canisters.

4.4.2. 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 percent RH, approximately 25°C, and 64 lpm continuous air flow containing 500 ppm HCHO for chin style canisters.

4.4.3. 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 percent RH, approximately 25°C, and 64 lpm continuous air flow containing 500 ppm HCHO for chin style canisters.

4.5. FORMALDEHYDE BENCH TESTS FOR PAPR CARTRIDGES/CANISTERS

4.5.1. Resistances and airflows for tight fitting PAPR will be taken before and after each test. Airflows only for loose fitting PAPR will be taken before and after each test.

4.5.2. Three cartridges (or pairs of cartridges) will be tested at 115 lpm for tight fitting PAPR or 170 lpm for loose fitting PAPR hood or helmet continuous air flow, approximately 25°C, and 50 ± 5 percent RH with 100 ppm HCHO. Tight fitting only PAPR gas mask canisters will be tested at 115 lpm at 500 ppm HCHO.

4.5.3. Two cartridges (or pairs of cartridges) will be equilibrated by passing 115 lpm for tight fitting PAPR or 170 lpm for loose fitting PAPR hood or helmet, continuous air flow through them at approximately 25°C, and 25 percent RH for six hours. They will then be tested at 115 or 170 lpm continuous air flow, approximately 25°C, 25 ± 5 percent RH, 100 ppm HCHO. Tight fitting only PAPR gas mask canisters will be tested at 115 lpm at 500 ppm HCHO.

4.5.4. Two cartridges (or pairs of cartridges) will be equilibrated by passing 115 lpm for tight fitting PAPR or 170 lpm for PAPR loose fitting hood or helmet, continuous air flow through them at 25°C, and 85 percent relative humidity for six hours. They will then be tested at 115 or 170 lpm continuous air flow, 25°C, 85 ± 5 percent RH, 100 ppm HCHO. Tight fitting only PAPR gas mask canisters will be tested at 115 lpm at 500 ppm HCHO.

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

5.2. Set up the VICI Metronics permeation system with the certified 1 ppm permeation tube for analyzer calibration.

5.2.1 Follow the instruction manual for the permeation system with regard to the proper dilution air flow rate, preconditioning of the permeation tube, and the correct temperature setting. Preconditioning and calibration may take 48-72 hours before testing of the respirator can begin.

5.2.2 Once the concentration has been determined from the permeation system, insert the intake tubing from the analyzer into the stream outlet of the permeation system.

5.2.3 Wait till the reading stabilizes, and adjust the span control to read the predetermined calculated concentration of the permeation tube.

5.3. Prepare solutions:

5.3.1. 10% Paraformaldehyde solution: In a 600 ml beaker containing 400 ml distilled water and a stirring bar, add 40g paraformaldehyde. Stir and heat the solution to 80°C using the heated magnetic stirrer. Place a watch glass over the top of the beaker or cover with aluminum foil to prevent excessive water loss. Continue stirring and heating until the solution clarifies (about 8-12 hours). Store in covered bottle or flask.

5.3.2. 1% starch: Weigh 1g starch and add to 100 ml boiling distilled water. Continue boiling until starch has dissolved. Discard when solution becomes cloudy.

5.3.3. 0.01N Iodine: Pipet 10 ml of 0.1N iodine to a 100 ml flask. Fill to mark with distilled water and invert 10-12 times to ensure complete mixing. Store in amber polyethylene bottle.

5.3.4. 1% Sodium Bisulfite: Weigh out 5g sodium bisulfite and dissolve in 500 ml distilled water. Store in polyethylene bottle.

5.3.5. Sodium Carbonate Buffer: Weigh out 40g sodium carbonate and transfer it to a 1 liter beaker containing a magnetic stir bar. Place the beaker on a magnetic stirrer. Add 250 ml of distilled water and stir until fully dissolved. Slowly add 10 ml glacial acetic acid while stirring. Dilute to 500 ml. Store in polyethylene bottle.

5.3.6. 0.1N Sodium Thiosulfate: Dissolve 24.82g sodium thiosulfate in 1 liter of distilled water.

5.3.7 0.01N Iodine: Pipet 10 ml of 0.1N iodine to a 100 ml flask. Fill to mark with distilled water and invert 10-12 times to ensure complete mixing. Store in amber polyethylene bottle.

5.4 Set up test equipment as shown in Figure 1. In addition to the humidity reading controlled by the Miller Nelson system, the Vaisala HMI 31 humidity indicator sensor is inserted into the air stream via a tee set-up directly prior to the introduction of the gas. This set up is not shown on Figure 1. The humidity reading obtained at this point takes into account tubing length and outside hood air temperature.

5.5. Determine the rate of advance of the syringe pump for the appropriate syringe volume, airflow, and the desired concentration for the respirator being tested. (See Tables 1 and 2.) Concentration will vary with the syringe volume size. The rate of the syringe pump must be recalculated when different size syringes are used or the airflow rate or concentration changes.

5.6. Turn on:

5.6.1. Miller Nelson Unit.

5.6.2. Air and water supplies.

5.6.3. Heated vaporizing element.

5.7. Establish the test concentration for approximately 100 ppm HCHO (500 ppm for canister testing.)

5.8. Fill syringe with 10% paraformaldehyde solution and place on the syringe pump. Set dial to calculated setting and start delivery into the flask. Allow 30-40 minutes for equalization of concentration. Make sure formaldehyde is delivered to the heated vaporizing element.

5.9. Add 10 ml 1% sodium bisulfite solution to the impinger.

5.10. Attach Gil-Air 3 sampling pump to intake side of the impinger. Connect outlet side of bubbler to Gilibrator. Check 1 lpm flow of the pump pulling through the sodium bisulfite solution. This setting will be used to sample the formaldehyde concentration. Remove outlet side from Gilibrator.

5.11. Connect tubing from the sample side of the impinger into the Gil-Air pump and tubing from the inlet side of the gas bubbler into the test concentration.

5.12. Turn on sampling pump and sample for 30 minutes at 1 lpm.

5.13. Shut off vacuum pump and transfer the contents of the impinger into an Erlenmeyer flask. Rinse the impinger stem and body and transfer the washings to the flask.

5.14. Place a stir bar in the flask and place the flask on the magnetic stirrer. Begin stirring.

5.15. Add 1 ml starch solution.

5.16. Add 0.1N Iodine dropwise until solution is dark blue.

5.17. Add 0.1N sodium thiosulfate dropwise until blue color is gone indicating neutralization of the excess iodine.

5.18. Slowly add 0.01N iodine dropwise from a 50 ml. buret until the first appearance of a faint blue color.

5.19. Add 25 ml sodium carbonate buffer solution. The blue color should disappear if formaldehyde is present.

5.20. Record the initial buret reading of the 0.01N Iodine.

5.21. Slowly add the 0.01N Iodine until the appearance of a faint blue color.

5.22. Record the final buret reading.

5.23 Calculate the concentration of formaldehyde using the formula in Table 3. Repeat steps 5.9 through 5.23 if concentration required is not obtained.

5.24. Once the test concentration has been established, testing may begin.

5.25. Weigh the cartridge or canister and record the weight.

5.26. Take inhalation and exhalation resistances of the cartridge or canister mounted on the facepiece at 85 Lpm. See Sections 84.122, 84.203, 84.1157, Title 42, Code of Federal Regulations, Part 84 for breathing resistance requirements. Take airflows of PAPR cartridge or canister mounted on blower assembly.

5.27. Mount cartridge or canister onto test fixture and place in testing chamber.

5.28. Direct challenge concentration airflow into test chamber. Start timer. Mount small piece of tygon tubing onto the outlet of the test fixture. Insert intake tubing of detector into a slit cut into the side wall of the tubing to allow the detector to sample at the flow rate of the detector without interference from airflow back pressure. Monitor and record upstream and downstream temperatures throughout testing. Record breakthrough values and times.

5.29. Run test until breakthrough of 1.0 ppm is observed or minimum service life is surpassed depending on type of cartridge or canister tested.

5.30. Dismount cartridge or canister, weigh and record final weight, and take final inhalation and exhalation resistances and PAPR airflows.

5.31. Turn off syringe pump and heater.

5.32. Allow clean air to purge through system for 10 - 15 minutes.

5.33. Turn off air and water supply to Miller Nelson system.

5.34. Turn off Miller Nelson system and analyzer.

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), Subpart I, Section 84.110(c), and Subpart L, Section 84.190(b); 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.

(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 Respirator 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.

84.190 Chemical cartridge respirators: description.

(b) Chemical cartridge respirators for respiratory protection against gases or vapors, which are not specifically listed with their maximum use concentration, may be approved if the applicant submits a request for such approval, in writing, to the Institute. The Institute shall consider each such application and accept or reject the application after a review of the effects on the wearer's health and safety and in the light of any field experience in use of chemical cartridge respirators as protection against such hazards.

7. RECORDS/TEST SHEETS

7.1. All test data will be recorded on the FORMALDEHYDE 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 Table 1- Calculation for syringe pump injection rates.

8.2 Table 2 - Nominal injection rates for Sage Model 355.

8.3 Table 3- Calculation for formaldehyde concentration.

8.4 Figure 1- Bench Top Set-Up.

8.5 Data Sheet.

TABLE 1

CALCULATIONS FOR SYRINGE PUMP INJECTION RATES at 25°C and 1 atm

For liquids:

$$C = \frac{(24.6 \times 10^6)\ KRp}{QM}$$

$$R = \frac{CQM}{(24.6 \times 10^6)\ KpX}$$

where:

R= rate of advance (mm/min) C = concentration (ppm)
K= syringe constant (ml/mm) p = solvent density (g/ml)
Q= airflow rate (lpm) M = molecular weight (g/mol)
X= decimal percent parformaldehyde solution (weight/volume)

To obtain the syringe constant, divide the volume of the syringe in ml by the length of the syringe in mm.

Sample calculation: Find the rate of advance and % of flow setting required to produce a concentration of 100 ppm HCHO in 64 lpm of air, using a 50 ml syringe and a 1.16 percent paraformaldehyde solution.

$$R = \frac{(100\ ppm)\ (64\ lpm)\ (30.03 g/mol)}{(24.6 \times 10^6)\ (0.620\ ml/mm)\ (1.0\ g/ml)(0.1116)}$$

R= 0.1129 mm/min.

From Table 2, the flow setting would be 800 at a range setting of 1/1000.

For syringe pumps delivering in volumes of milliliters/minute, multiply rate of advance R by syringe constant.

ml/min.= 0.1129 mm/min. x 0.620 ml/min.
ml/min.= .070

TABLE 2

NOMINAL INJECTION RATES FOR SAGE SYRINGE PUMP MODEL 355

	Rate of advance (R) (mm/min.)			
% Flow dial setting	Range setting 1	1/10	1/100	1/1000
100	14.5	1.45	0.145	0.0145
150	21.8	2.18	0.218	0.0218
200	29.0	2.90	0.0290	0.0290
250	36.3	3.63	0.0363	0.0363
300	43.5	4.35	0.0435	0.0435
350	50.8	5.08	0.508	0.0508
400	58.0	5.80	0.580	0.0580
450	65.3	6.53	0.653	0.0653
500	72.5	7.25	0.725	0.0725
550	79.8	7.98	0.798	0.0798
600	87.0	8.70	0.870	0.0870
650	94.3	9.43	0.943	0.0943
700	102	10.2	1.02	0.102
750	109	10.9	1.09	0.109
800	116	11.6	1.16	0.116
850	123	12.3	1.23	0.123
900	131	13.1	1.31	0.131
950	138	13.8	1.38	0.138
1000	145	14.5	1.45	0.145

TABLE 3

CALCULATION OF FORMALDEHYDE CONCENTRATION

The formaldehyde equivalent F_e is given by the formula:

$$F_e = \frac{(N)\,I}{.01} \times 0.15 \text{ mg.}$$

Where (N) I is the normality of the standardized iodine. This formula is based on the fact that the equivalent weight of formaldehyde is 15 gm., which means that 1 ml. of 0.01N iodine is equivalent to 0.15 mg. of formaldehyde.

The amount of formaldehyde in the sample volume collected is given by the formula:

F_c = final buret reading - initial buret reading x $F_e$mg

F_c = total ml. .01 iodine used x 0.15 mg

Formaldehyde concentration in ppm is given by the following formula:

$$\text{ppm} = \frac{F_c}{V} \times \frac{24.45}{MW} \times \frac{760}{P} \times \frac{T+273}{298} \times 10^3$$

Where:

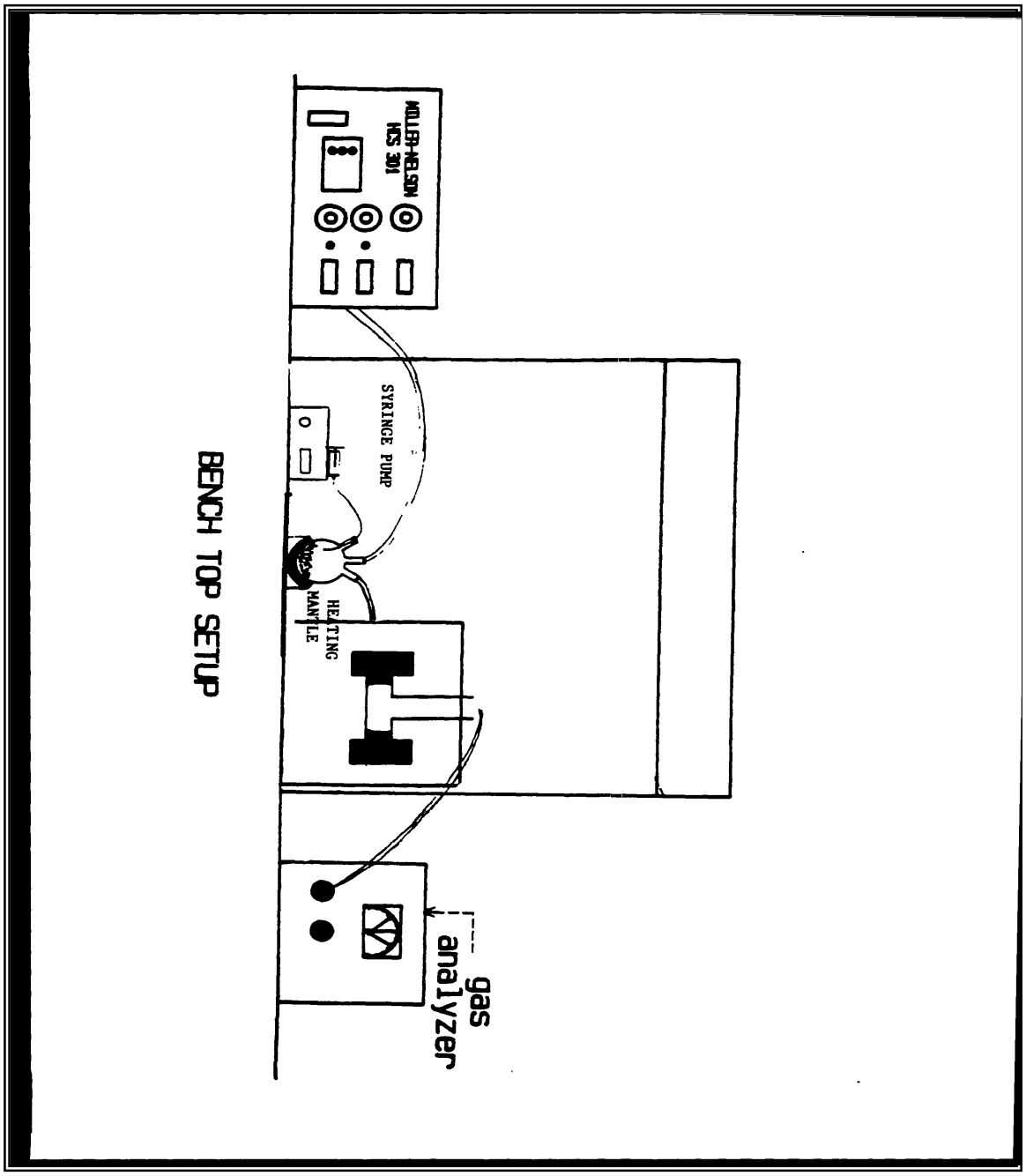
V = volume of sample collected (30 Lpm)
MW = molecular weight formaldehyde (30.03 g/mole)
P = pressure in mm of mercury in the lab
T = temperature in °C of the lab

Assuming that T is equal to 25°C and p is equal to 760 mm mercury, the ppm formula simplifies to:

HCHO (ppm) = F_c x 27.14 assuming volume of sample collected 30 liters

HCHO (ppm) = ml. 0.01 Iodine used x 0.15 mg x 27.14

Figure 1- Bench Top Set-Up



NIOSH *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
	Inhalation		Exhalation		Inhalation		Exhalation		
Test			Initial		Initial	Final	Initial	Final	
1									
2									
3									
4									
5									
6									
7									

Overall Results: Pass _____ Fail _____ Comment:

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

Overall Results: Pass _____ Fail _____ Comment:

Was all testing equipment in calibration throughout all testing: Yes _____ No

Signature: _____ Date:

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