National Institute for Occupational Safety and Health National Personal Protective Technology Laboratory P.O. Box 18070 Pittsburgh, PA 15236		
Procedure No. RCT-APR-STP-0062	Revision: 1.1	Date: 6 September 2005

DETERMINATION OF NITROGEN DIOXIDE 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 nitrogen dioxide service life requirements on chemical cartridge, 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 L, Section 84.190; Volume 60, Number 110, June 8, 1995.

2. GENERAL

This STP describes the Determination of Nitrogen Dioxide 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.

Approvals:	1st Level	2nd Level	3rd Level

3.1.2. 

Interscan Corporation Model 1152 Nitrogen Dioxide Analyzer or equivalent.

3.1.3. Nitrogen dioxide (NO_2) cylinder, 99% purity with 61A-660 regulator.

3.1.4. 

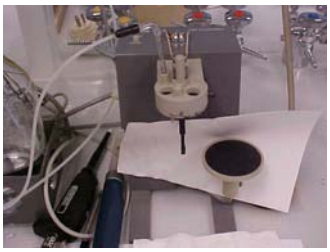
Nitrogen dioxide certified gas cylinder, 1 ppm NO_2, balance gas helium with 3510-660 regulator.

3.1.5. 

Brooks Rotameters, R 215-D for cartridges or mass flow controller, 0-50 ml/min.

3.1.6. 

Digi Sense Scanning Thermocouple Thermometer Model 92800-10.

3.1.7.  
Radiometer America Multi-Titration System, Model DTS 800, or equivalent.

3.1.8. 
"The Gilbrator", Primary Standard Airflow Calibrator, or equivalent.

3.1.9. 

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

3.1.10. Water bath, cylinder wrap, or room heater, regulated to provide cylinder with constant 80-85^{o}F.

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

3.1.12. 
Erlenmeyer flasks, 250 to 500 milliliters (mL).

3.1.13. 
Pipets, 5 mL.

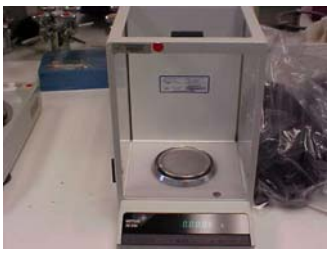
3.1.14. Potassium Iodide (KI) (granular).

3.1.15. Potassium Iodate (KIO_3) (granular).

3.1.16. Starch, Soluble Potato, Powder.

3.1.17. Boric Acid (granular).

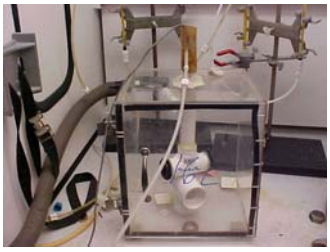
3.1.18. Sodium Thiosulfate ($Na_2S_2O_3$) (granular).

3.1.19. 
Electronic Balance with an accuracy of 0.001 grams (g).

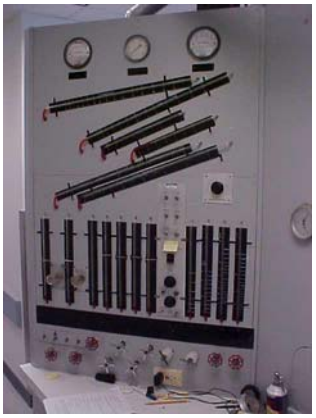
3.1.20. 
Houston Instruments, Model 451BE, strip chart recorder or equivalent.

3.1.21. Test fixture for holding cartridges. The test fixture is specific to each

manufacturer depending on how the cartridge is mounted to the facepiece. In most cases the cartridge cups of the respirator are affixed by hot melt glue to a PVC pipe tee of appropriate size. The T end has a 29/42 ground glass joint glued in place.

3.1.22. 

Test chamber consisting of a 12" x 11½" x 7" air tight box, made of ½" plexiglass with two hinge type locks on the door opening lined with gasket material. A ½" hole is located on the back side 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.

3.1.23. 

Resistance tester consisting of a vacuum source capable of delivering 85 liters per minute (lpm), a 6-inch slant manometer, a 29/42 female ground glass joint. The resistance testers currently being used are located on the silica dust and lead fume chambers.

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.

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. NITROGEN DIOXIDE 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 ± 5% relative humidity (RH), approximately 25 degrees Celsius (°C), and 500 ppm of NO_2.

4.3.3. Two cartridges or pairs of cartridges will be equilibrated at room temperature by passing 25% RH air through them at 25 lpm for 6 hours and then testing them at 25% RH, approximately 25°C, and 64 lpm continuous flow rate containing 500 ppm NO_2.

4.3.4. Two cartridges or pairs of cartridges will be equilibrated at room temperature by passing 85% RH air through them at 25 lpm for 6 hours and then testing them at 85% RH, approximately 25°C, and 64 lpm continuous flow rate containing 500 ppm NO_2.

4.4. **Please refer to Material Safety Data Sheet 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. After the manufacturer's specified warmup period, calibrate the NO_2 analyzer using the certified gas cylinder containing the 1 ppm standard as follows:

5.2.1. With a tee in line on the gas cylinder, insert the intake tubing into the tee.

5.2.2. Turn on the cylinder.

5.2.3. Wait till reading stabilizes, and adjust span control to read 1 ppm.

5.3. Prepare solutions:

5.3.1. 5% Potassium Iodide/1% Potassium Iodate: Dissolve 50 g KI and 10g KIO_3 in 1 liter distilled water.

5.3.2. 0.0025 Normal (N) Sodium Thiosulfate Solution: Dissolve 0.6205 g sodium thiosulfate in 1 liter of distilled water. Commercially bought certified sodium thiosulfate solutions can be used.

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

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

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

5.3.3.3. Discard when starch solution becomes cloudy.

5.4. Connect the Gil-Air-3 Sampling Pump to the Gillibrator and calibrate to draw 1 lpm. This will be used to draw and collect the test concentration sample onto the KI/KIO_3 solution.

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

5.6. Heat cylinder to 80-85^{o}F to force nitrogen dioxide gas from cylinder.

5.7. Turn on:

5.7.1. Miller Nelson Unit.

5.7.2. Air and water supplies.

5.7.3. NO_2 cylinder.

5.7.4. NO_2 analyzer.

5.8. Establish test concentration for approximately 500 ppm NO_2.

5.9. Measure 100 mL of the 1%KIO_3/5% KI solution into the gas bubbler. Attach Gil-Air 3 sampling pump to intake side of the gas bubbler. Connect outlet side of bubbler to Gillibrator. Check 1 lpm flow of the pump pulling through the 1%KIO_3/5% KI solution.

5.10. Adjust the rotameter or flow controller to deliver approximately 32 cc/min. NO_2 into the airstream to obtain 500 ppm NO_2.

5.11. Connect sample side of gas bubbler into the Gil-Air-3 Sampling Pump and intake into the test gas concentration.

5.12. Turn pump on and sample at 1 lpm for 10 minutes. The KI/KIO_3 solution should change to dark yellow in the presence of nitrogen dioxide.

5.13. Remove gas bubbler, and transfer the solution into an Erlenmeyer flask.

5.13.1. Rinse the gas bubbler with distilled water and transfer the washings into the flask.

5.14. Titrate the solution with 0.0025N sodium thiosulfate until it is pale yellow.

5.15. Add 5 mL of starch indicator. Solution will turn dark blue.

5.16. Continue titration until the blue color just disappears.

5.17. Calculate the concentration of NO_2 in air using the following formula:

$$9.168 \times \text{mL of .0025N } Na_2S_2O_3 = \text{ppm } NO_2$$

5.18. Once the NO_2 concentration has been established testing may begin.

5.19. Weigh the cartridge and record the weight.

5.20. Take inhalation and exhalation resistances of the cartridges mounted on the facepiece at 85 lpm. See Section 84.203 Title 42, Code of Federal Regulations, Part 84 for breathing resistance requirements.

5.21. Mount cartridge onto test fixture and place in testing chamber.

5.22. Direct challenge concentration airflow into test chamber. Start timer. Mount small piece of tygon tubing onto outlet of test fixture. Insert intake tubing of detector into side wall of tygon 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.

5.23. Run test until breakthrough of 1.0 ppm is observed or minimum service life is surpassed.

5.24. Dismount cartridge or canister, weigh and record final weight, and take final inhalation and exhalation resistances.

5.25. Turn off nitrogen dioxide cylinder.

5.26. Allow clean air to purge system for 15 minutes. Disconnect NO_2 tubing from test system to prevent humidity sensor contamination.

5.27. Turn off air and water supply.

5.28. Turn off Miller Nelson System.

5.29. Turn off nitrogen dioxide detector.

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 L, Section 84.190; 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.

84.190 Chemical cartridge respirators: description.

(a) Chemical cartridge respirators including all completely assembled respirators which are designed for use as respiratory protection during entry into or escape from atmospheres not immediately dangerous to life and health, are described according to the specific gases or vapors against which they are designed to provide respiratory protection, as follows:

Type of chemical cartridge respirator[1]	Maximum use concentration, parts per million
Ammonia	300
Chlorine	10
Hydrogen chloride	50

Methyl amine	100
Organic vapor	[2]1,000
Sulfur dioxide	50
Vinyl chloride	10

[1]Not for use against gases or vapors with poor warning properties (except where MSHA or Occupational Safety and Health Administration standards may permit such use for a specific gas or vapor) or those which generate high heats of reaction with sorbent material in the cartridge.
[2]Maximum use concentrations are lower for organic vapors which produce atmospheres immediately hazardous to life or health at concentrations equal to or lower than this concentration.

(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.

6.3 The criterion for passing this test is that the penetration of NO_2 shall not exceed 1.0 ppm for a minimum service life of 30 minutes (Standard SO_2 test time) for negative pressure cartridges. If the penetration does not exceed 1.0 ppm during the service life of 30 minutes for negative pressure cartridges, the units pass the test. If the penetration does exceed 1.0 ppm for a minimum service life of 30 minutes for negative pressure cartridges, the units fail the test. The units will be run until the penetration of NO_2 is equal to 1.0 ppm.

7. RECORDS/TEST SHEETS

7.1. All test data will be recorded on the NITROGEN DIOXIDE 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. List of Test Equipment Manufacturers.

8.2. Test Data Sheet

Attachment 8.1.

8.1.1. Miller Nelson Research
8 Harris Court
Monterey, CA 93940
408-647-1551

8.1.2. Interscan Corporation
21700 Nordhoff St.
Chatsworth, CA 91311

8.1.3. Matheson Gas Products
1650 Enterprise Parkway
PO Box 358
Twinsburg, OH 44087

8.1.4. Brooks Instrument Div.
Emerson Electric Co.
Hatfield, PA 07630

8.1.5. Radiometer America
811 Sharon Drive
Westlake, OH 44145

8.1.6. Gilian Instrument Corp.
35 Fairfield Place
W. Caldwell, NJ 07006

8.1.7. Fisher Scientific
585 Alpha Drive
Pittsburgh, PA 15338

8.1.8. Cole Parmer Instrument Co.
7425 North Oak Park Avenue
Niles, IL 60714

Attatchment 8.2

National Institute for Occupational Safety and Health
Respirator Branch
Test Data Sheet

NIOSH

Task Number: TN-9734
Test: Nitrogen Dioxide Test
Manufacturer: Aearo Corporation
Item Tested: R610 cartridge

Reference No.: CFR 84.190
STP No.: 62

RESISTANCE

Respirator Type: Cartridge only for single type of gas

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	40	45	20	20	28.4	31.8	11.4	11.4	PASS
2	40	45	20	20	27.9	31.0	11.4	11.4	PASS
3	40	45	20	20	27.4	25.9	11.4	11.4	PASS
4	40	45	20	20	28.4	29.2	11.4	11.4	PASS
5	40	45	20	20	28.4	29.2	11.4	11.4	PASS
6	40	45	20	20	27.4	27.9	11.4	11.4	PASS
7	40	45	20	20	27.9	28.4	11.4	11.4	PASS

Overall Result: PASS

WEIGHTS (gm.) Test	As Received	Pre-Conditioned	Water Gain	Final Weight	Concentration (ppm)	AIR FLOW (Lpm) RH%	Minimum Allowable	Initial	Final	Result
1	193.1			202.4	500	50	0	0	0	
2	191.8			197.7	500	50	0	0	0	
3	191.9			197.7	500	50	0	0	0	
4	190.6	221.1	30.5	224.8	500	85	0	0	0	
5	192.4	223.1	30.7	224.1	500	85	0	0	0	
6	192.8	192.9	0.1	195.5	500	25	0	0	0	
7	191.9	192	0.1	196.1	500	25	0	0	0	

Overall Result:

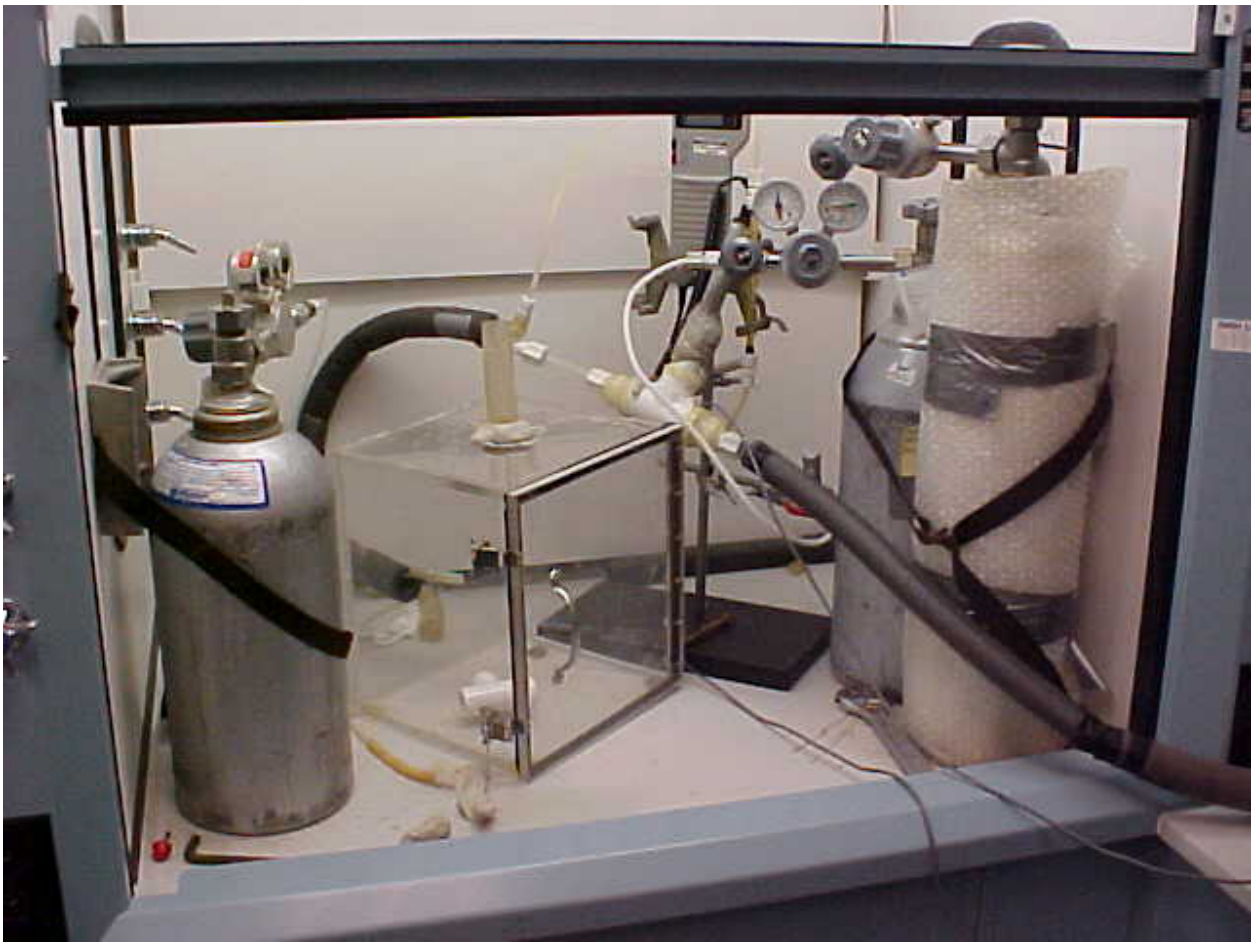
DATA TABLE

Test	Test Condition	Breakthrough (ppm)	Temperature (C) Dnstream	Temperature (C) Upstream	Minimum Service Life (min.)	Final Time (min.)	Result
1	As Received	0.00	28.2	25.1	30	42.6	PASS
2	As Received	0.00	28.1	25.0	30	36.9	PASS
3	As Received	0.30	28.6	25.1	30	33.5	PASS
4	85% Preconditioned	0.40	26.6	25.0	30	33.5	PASS
5	85% Preconditione	0.30	26.8	24.9	30	37.0	PASS
6	25% Preconditioned	0.35	26.8	25.1	30	41.9	PASS
7	25% Preconditioned	0.40	26.7	25.0	30	38.2	PASS

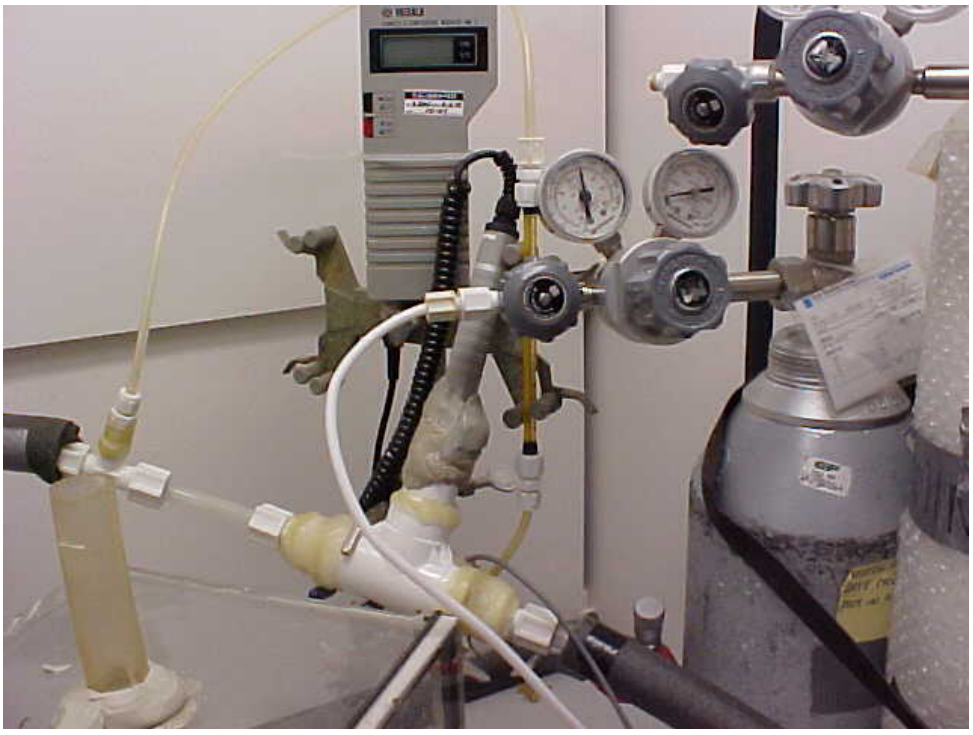
Overall Result: PASS

Signature: Cathy Calvert
Engineering Technician

Date: 1/16/98




MNR



Revision History

Revision	Date	Reason for Revision
1.0	5 June 2000	Historic document
1.1	6 September 2005	Update header and format to reflect lab move from Morgantown, WV No changes to method