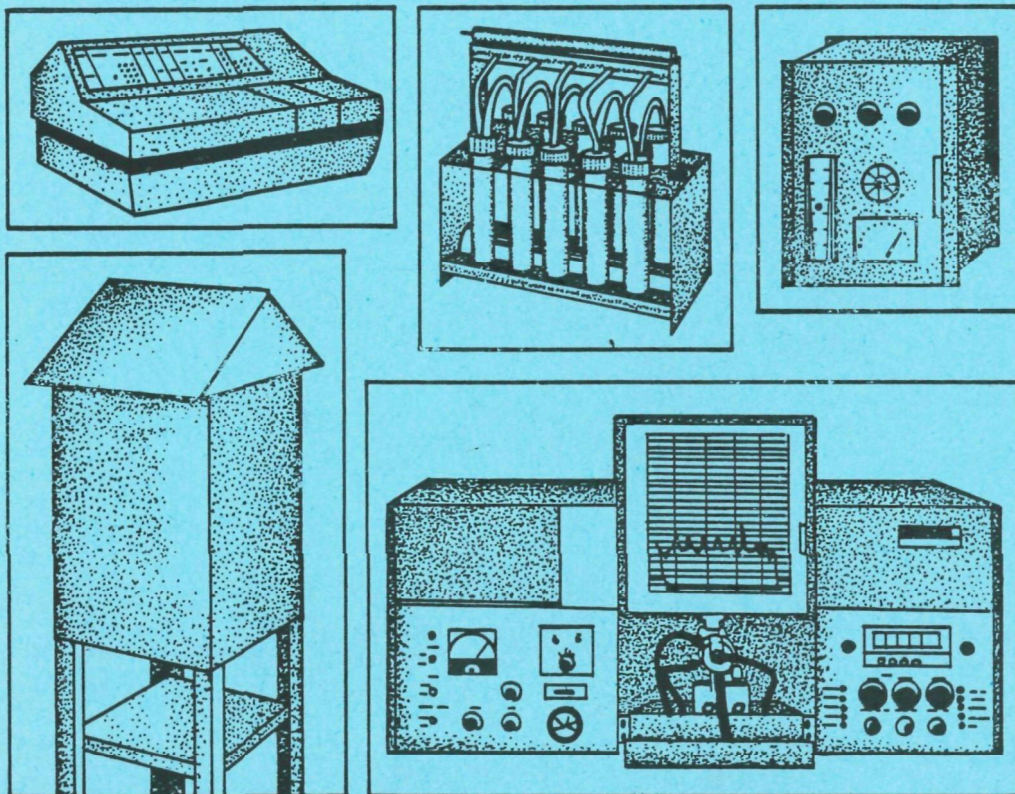**♻EPA**

APTI
Correspondence Course 438
Reference and Automated
Equivalent Measurement
Methods for Ambient Air
Monitoring

Guidebook

Air

APTI
Correspondence Course 438
Reference and Automated Equivalent Measurement Methods for Ambient Air Monitoring

Guidebook

Notice

This is not an official policy and standards document. The opinions and selections are those of the authors and not necessarily those of the Environmental Protection Agency. Every attempt has been made to represent the present state of the art as well as subject areas still under evaluation. Any mention of products or organizations does not constitute endorsement by the United States Environmental Protection Agency.

Table of Contents

Course Introduction

Overview of Course

Course Description

This training course is a thirty-hour correspondence course concerning EPA-designated reference and equivalent measurement methods for ambient air quality monitoring. Automated nonmethane organic compound analyzers are also discussed. Course topics include the following:
- reference and automated equivalent measurement methods for sulfur dioxide
- reference measurement methods for ozone
- reference measurement method for total suspended particulate matter
- reference measurement methods for carbon monoxide
- reference measurement methods for nitrogen dioxide
- reference measurement method for lead
- automated analyzers for nonmethane organic compounds.

Course Goal

To familiarize you with EPA-designated reference and automated equivalent measurement methods for ambient air quality monitoring and air quality analyzers for nonmethane organic compounds.

Course Objectives

After completing this course, you should be able to—
1. describe the manual reference measurement methods for sulfur dioxide, total suspended particulate matter, and lead, and recognize considerations for the operation, maintenance, calibration, and auditing of equipment used in the methods; and
2. describe considerations for the operation, maintenance, calibration, and auditing of air quality analyzers for sulfur dioxide, ozone, carbon monoxide, nitrogen dioxide, and nonmethane organic compounds.

Sequence, Section Titles, and Trainee Involvement Time

Section number	Section title	Trainee involvement time (hours)
1	Reference Method for Sulfur Dioxide	3
2	Automated Equivalent Measurement Methods for Sulfur Dioxide	3½
3	Reference Methods for Ozone	3½
	Quiz 1	½
4	Reference Method for Total Suspended Particulate Matter (TSP)	4
5	Reference Methods for Carbon Monoxide	3
6	Reference Methods for Nitrogen Dioxide	3
	Quiz 2	½
7	Reference Method for Lead	3½
8	Automated Analyzers for Nonmethane Organic Compounds	4
	Final Exam	1½

Requirements for Successful Completion of this Course

In order to receive three Continuing Education Units (CEUs) and a certificate of course completion you must fulfill the following requirements:
- take two supervised quizzes and a supervised final examination
- achieve a final course grade of at least 70 (out of 100) determined as follows:
  - 20% from Quiz 1
  - 20% from Quiz 2
  - 60% from the final examination

Use of Course Materials

Necessary Materials

- *APTI Correspondence Course 438 Reference and Automated Equivalent Measurement Methods for Ambient Air Monitoring: Guidebook*
- EPA 450/2-81-018b, *APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual, Second Edition*
- EPA 600/4-81-015, *Technical Assistance Document for the Calibration and Operation of Automated Ambient Non-Methane Organic Compound Analyzers*
- pencil or pen
- calculator (optional, but recommended)

Use of this Guidebook

Relationship Between Guidebook and Assigned Reading Materials

This guidebook directs your progress through the reference texts *APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual, Second Edition* and *Technical Assistance Document for the Calibration and Operation of Automated Non-Methane Organic Compound Analyzers* and through the excerpts of Section 2.8 of *Quality Assurance Handbook for Air Pollution Measurement Systems*, which are contained in this guidebook.

Description of Guidebook Sections

This guidebook contains reading assignment sections that correspond to lessons of the course.

Each section contains the following:
- reading assignment
- section's learning goal and objectives
- reading guidance
- review exercise

Please *do not* write in this guidebook.

Instructions for Completing the Quizzes and Examinations

- You should have received, along with this guidebook, a separate *sealed* envelope containing two quizzes and a final examination.
- You must arrange to have someone serve as your test supervisor.
- You must give the *sealed* envelope containing the quizzes and examination to your test supervisor.
- At the designated times during the course, under the supervision of your test supervisor, complete the quizzes and the final exam.
- After you have completed each quiz or the exam, your test supervisor must sign a statement on the quiz/exam answer sheet certifying that the quiz/exam was administered in accordance with the specified test instructions.
- After signing the answer sheet, your test supervisor must mail the quiz/exam and its answer sheet to the following address:

  Air Pollution Training Institute
  Environmental Research Center
  MD 20
  Research Triangle Park, NC 27711

- After completing a quiz, continue with the course. Do *not* wait for quiz results.
- Quiz/exam and course grade results will be mailed to you.

If you have questions, contact the Air Pollution Training Institute:

Air Pollution Training Institute
Environmental Research Center
MD 20
Research Triangle Park, NC 27711

Telephone numbers:
        Commercial: (919) 541-2401
        FTS: 629-2401

Section 1

Reference Method for Sulfur Dioxide

Reading Assignment

Pages 1-1 through 2-12 of EPA 450/2-81-018b *APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual.*

Reading Assignment Topics

- Introduction to reference and equivalent measurement methods
- Reference method for sulfur dioxide

Learning Goal and Objectives

Learning Goal

The purpose of this section is to familiarize you with general information about reference and equivalent measurement methods and with specific information concerning the sulfur dioxide reference method.

Learning Objectives

At the end of this section, you should be able to—

1. identify pollutants having National Ambient Air Quality Standards (NAAQS),
2. define primary and secondary NAAQS,
3. locate, in the *Code of Federal Regulations*, the NAAQS, descriptions of the reference methods and reference measurement principles, and designation requirements for reference and equivalent methods,
4. identify the two general types of reference and equivalent methods,
5. describe requirements for the designation of automated reference methods and for the designation of manual and automated equivalent methods,
6. recognize sampling and analytical equipment used in the sulfur dioxide reference method,
7. recognize two chemical reactions involved in the sampling and analysis of sulfur dioxide by the reference method,
8. describe the sodium sulfite and permeation tube calibration methods for the sulfur dioxide reference method,
9. identify and describe the removal of three potential interferences in the sulfur dioxide reference method,
10. recognize and describe solutions for eight reagent problems of the sulfur dioxide reference method, and

11. describe three types of audits that should be included in a quality assurance program for the sulfur dioxide reference method.

Reading Guidance

- When you have finished the reading assignment, complete the review exercise for Section 1. It begins on the following page.
- After you have answered the review exercise questions, check your answers. The correct answers are listed on the page immediately following the review exercise.
- For any review exercise questions that you answered incorrectly, review the page(s) of the reading assignment indicated on the answers page.
- After you have reviewed your incorrect answers (if any), proceed to Section 2 of this guidebook.

Review Exercise

Now that you've completed the assignment for Section 1, please answer the following questions. These will help you determine whether you are mastering the material.

For each of questions 1 through 3, match the item with its location in the *Code of Federal Regulations*.

1. National Ambient Air Quality Standards (NAAQS)
2. descriptions of the reference methods and reference measurement principles for monitoring pollutants that have NAAQS
3. designation requirements for reference and equivalent methods

   a. Title 40 Part 53
   b. Title 40 Part 50
   c. Appendixes of Title 40 Part 50

4. Which of the following is a(are) general type(s) of reference methods?
   a. manual method
   b. automated method
   c. both a and b, above
   d. none of the above

5. Which of the following is a(are) general type(s) of equivalent methods?
   a. manual method
   b. automated method
   c. both a and b, above
   d. none of the above

6. True or False? In order to be designated an equivalent method, a manual method must demonstrate a consistent relationship to the manual reference method or to the automated reference methods.

7. Which of the following is(are) necessary for the designation of an automated equivalent method?
   a. Performance specifications for automated methods must be met.
   b. Consistent relationship requirements must be met.
   c. both a and b, above
   d. none of the above

8. Which of the following is(are) necessary for the designation of an automated reference method?
   a. Performance specifications for automated methods must be met.
   b. The reference measurement principle must be used.
   c. both a and b, above
   d. none of the above

9. National Ambient Air Quality Standards (NAAQS) have been established for which of the following?
   a. sulfur dioxide
   b. total suspended particulate matter
   c. mercury
   d. both a and c, above
   e. both a and b, above

10. Which of the following has no NAAQS?
    a. nitric oxide
    b. nitrogen dioxide
    c. lead
    d. ozone

11. Primary NAAQS are established to protect public ____(?)____.
    a. health
    b. welfare
    c. health and welfare
    d. none of the above

12. Secondary NAAQS are established to protect public ____(?)____.
    a. health
    b. welfare
    c. health and welfare
    d. none of the above

For each of questions 13 through 17, match the chemical with its use in the reference method for sulfur dioxide.

13. formaldehyde

14. pararosaniline

15. potassium tetrachloromercurate (TCM)

16. sulfamic acid

17. EDTA/phosphoric acid

a. color development of sample solution

b. complexing of sulfur dioxide in absorbing solution

c. destruction of nitrite in sample solution

d. removal of heavy metal interference

18. True or False? Initial and final sample air flow rates of sulfur dioxide sampling trains must be determined at the sampling sites.

19. Which of the following is a(are) component(s) of a 24-hour sampling train for sulfur dioxide?
    a. vacuum pump
    b. absorber tube
    c. moisture trap
    d. a and b, above
    e. a, b, and c, above

20. Which of the following devices may be used for controlling the flow of sample air through a sulfur dioxide sampling train?
   a. rotameter combined with a needle valve
   b. critical orifice
   c. spirometer
   d. a and b, above
   e. a, b, and c, above

21. True or False? A membrane particulate filter should be placed downstream of a critical orifice used in a sulfur dioxide sampling train.

22. In the sulfite solution calibration method for sulfur dioxide analysis, the standardized sulfite solution is diluted with ____(?)____.
   a. sodium thiosulfate
   b. iodine
   c. TCM
   d. starch solution

23. True or False? Reaction time and temperature must be carefully controlled during the analysis of sulfur dioxide samples and standard solutions.

24. As light of an appropriate wavelength passes through the sulfur dioxide solutions, the strength of the light beam ____(?)____ in direct proportion to a(an) ____(?)____ in the concentrations of the solutions.
   a. increases, increase
   b. decreases, increase
   c. increases, decrease

25. Under the conditions described below, the sulfur dioxide concentration measured by the sulfur dioxide reference method is ____(?)____ $\mu g/std\ m^3$.
   a. 0.3
   b. 3   Given: Absorbance of sample solution: 0.190 absorbance units
   c. 30   Absorbance of reagent blank: 0.160 absorbance units
   d. 300   Calibration factor (B_s): 31 μg/absorbance unit
                            Volume of absorber solution analyzed (V_a): 5 mL
                            Total volume of solution in absorber (V_b): 50 mL
                            Volume of air sampled: 310 L, at reference conditions
                               of 25 °C and 760 mm Hg

26. True or False? An SO_2 permeation tube consists of a tube that is partially filled with liquefied SO_2. The gaseous SO_2 above the liquid is able to permeate the walls of the tube.

27. Which of the following is an(are) important consideration(s) when using a permeation tube system?
   a. The permeation tube must be kept at a constant temperature.
   b. All gas flow rates must be measured accurately.
   c. Dilution air must be saturated with water vapor.
   d. only a and b, above
   e. only b and c, above

28. Under the conditions described below, what is the SO_2 concentration (μg/std m^3) generated by the permeation system?
    a. 0.2
    b. 20   Given: Permeation rate of SO_2 from tube: 1.000 μg/min
    c. 202   Flow rate of dilution air: 4.950 std L/min
    d. 200   Flow rate of inert carrier gas: 0.050 std L/min

29. True or False? A reagent blank should be analyzed when analyzing standard or sample solutions by the SO_2 reference method.

30. Which of the following eliminates ozone interference in the SO_2 reference method?
    a. a 20-minute time delay between sampling and analysis
    b. sulfamic acid
    c. EDTA
    d. formaldehyde

31. Under which of the following conditions should TCM reagent *not* be used for collecting SO_2?
    a. The pH of the TCM reagent is between 3 and 5.
    b. A precipitate is present in the TCM reagent.
    c. both a and b, above
    d. none of the above

32. A sulfur dioxide sample must be invalidated if the difference between its initial and final sampling standard flow rates is greater than ____(?)____ percent.
    a. 1
    b. 2
    c. 5
    d. 10

For each of questions 33 and 34, match the chemical with its characteristic.

33. TCM reagent   a. poisonous
34. monochlorosulfantomercurate complex   b. subject to decomposition by heat and sunlight

35. Which of the following is an(are) important consideration(s) concerning the spectrophotometer used in the SO_2 reference method?
    a. The band width is important and should be periodically checked.
    b. The absorbance cells should be washed after use to prevent staining.
    c. Absorbance readings should be made as quickly as possible.
    d. all of the above

36. True or False? Pararosaniline used in the SO_2 reference method must meet specifications described in the method.

37. True or False? All water used in preparing reagents for the SO_2 reference method must contain oxidants.

38. Which of the following activities should be included in a quality-assurance program for the SO_2 reference method?
    a. audits of sampler flow measuring devices
    b. analyses of analytical control samples
    c. data processing audits
    d. all of the above

39. True or False? Audits for the SO_2 reference method should be performed by persons not routinely involved in the sampling and analysis of SO_2.

40. In the SO_2 reference method, SO_2 reacts with _____(?)_____ to form a monochlorosulfantomercurate complex.
    a. sulfamic acid
    b. formaldehyde
    c. pararosaniline
    d. none of the above

41. In the SO_2 reference method, a monochlorosulfantomercurate complex reacts with pararosaniline and _____(?)_____ to form a pararosaniline methyl sulfonic acid.
    a. sulfamic acid
    b. formaldehyde
    c. EDTA
    d. none of the above

Section 1
Review Exercise Answers

Page(s) of *Analytical Methods*
for Air Quality Standards:
Student Manual

1. b 1-1
2. c 1-1
3. a 1-1
4. c 1-1
5. c 1-2
6. True 1-2
7. c 1-2
8. c 1-1
9. e 1-1
10. a 1-1
11. a 1-1
12. b 1-1
13. a 2-2
14. a 2-2
15. b 2-1
16. c 2-2
17. d 2-8
18. True 2-4
19. e...............................2-2, 2-3
20. d 2-2
21. False 2-3, 2-4
22. c 2-5
23. True 2-5
24. b 2-5
25. c 2-6
26. True 2-6
27. d 2-6
28. d 2-7
29. True 2-8
30. a 2-8
31. b 2-9
32. c 2-4
33. a 2-10
34. b 2-9
35. d 2-10
36. True 2-10

Section 2

Automated Equivalent Measurement Methods for Sulfur Dioxide

Reading Assignment

Pages 3-1 through 3-19 of EPA 450/2-81-018b *APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual.*

Reading Assignment Topics

- Amperometric instruments
- Flame photometric instruments
- Fluorescence instruments
- Second derivative spectroscopic instruments
- Conductimetric instruments

Learning Goal and Objectives

Learning Goal

The purpose of this section is to familiarize you with automated equivalent methods for monitoring sulfur dioxide.

Learning Objectives

At the end of this section, you should be able to—

1. identify two advantages of using continuous instrumental methods for monitoring ambient air quality;
2. describe measurement principles used by amperometric, flame photometric, fluorescence, second derivative spectroscopic, and conductimetric sulfur dioxide monitors;
3. recognize and identify advantages and disadvantages of conductimetric, amperometric, second derivative spectroscopic, flame photometric, and fluorescence sulfur dioxide monitors;
4. identify and describe the removal of six potential interferences associated with the use of amperometric, flame photometric, and fluorescence sulfur dioxide monitors;
5. describe calibration, maintenance, and trouble-shooting procedures for amperometric, flame photometric, fluorescence, second derivative spectroscopic, and conductimetric sulfur dioxide monitors;

6. recognize the need for frequent zero and span checks of automated air quality monitors; and

7. identify a quality-assurance reference text for automated ambient-air-quality monitors.

Reading Guidance

- Refer often to the figures of the assigned reading material as you progress through the assignment.
- When you have finished the reading assignment, complete the review exercise for Section 2. It begins on the following page.
- After you have answered the review exercise questions, check your answers. The correct answers are listed on the page immediately following the review exercise.
- For any review exercise questions that you answered incorrectly, review the page(s) of the reading assignment indicated on the answers page.
- After you have reviewed your incorrect answers (if any), proceed to Section 3 of this guidebook.

Review Exercise

Now that you've completed the assignment for Section 2, please answer the following questions. These will help you determine whether you are mastering the material.

1. Which of the following is an(are) advantage(s) of using continuous instrumental methods for monitoring ambient air quality?
   a. real-time data output
   b. ability to transmit data directly into computerized data acquisition and telemetry systems
   c. both a and b, above
   d. none of the above

For each of questions 2 through 6, match the instrumental measurement principle with its description.

2. amperometric
3. flame photometric
4. second-derivative spectroscopic
5. conductimetric
6. fluorescence

a. uses ion changes in an acidified hydrogen peroxide solution to measure gaseous pollutant concentration
b. uses an electrical current generated by oxidation-reduction reactions in an electrolytic cell to measure gaseous pollutant concentration
c. relates the slope and curvature characteristics of energy absorption bands to gaseous pollutant concentration
d. uses the energy emitted by a pollutant in a hydrogen-rich flame to measure the pollutant's concentration
e. uses the energy emitted by "excited molecules" (molecules having excess energy) to measure pollutant concentration

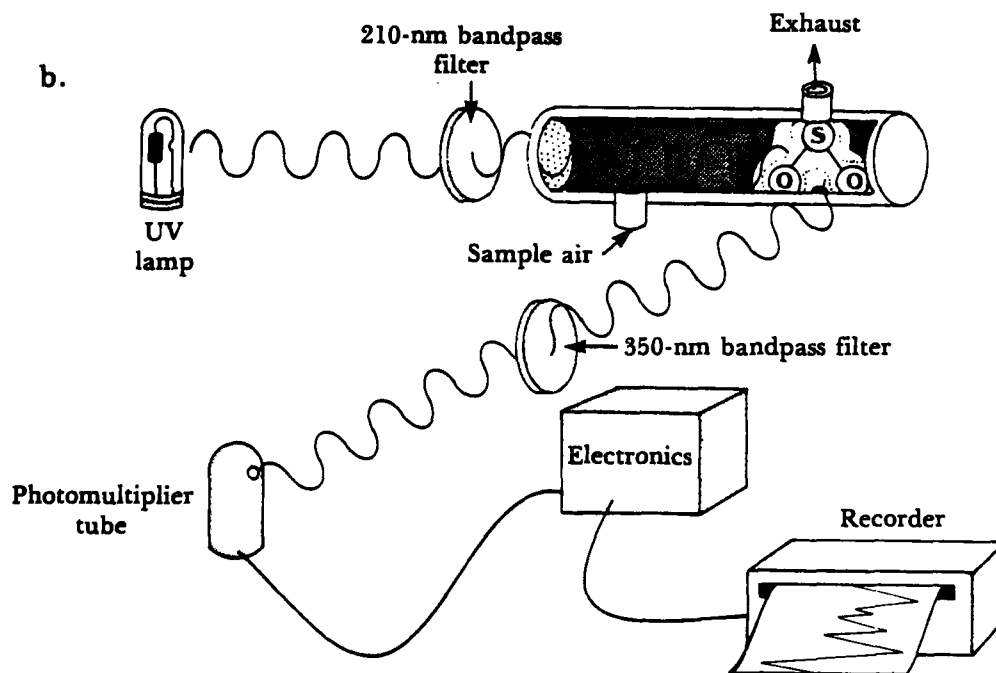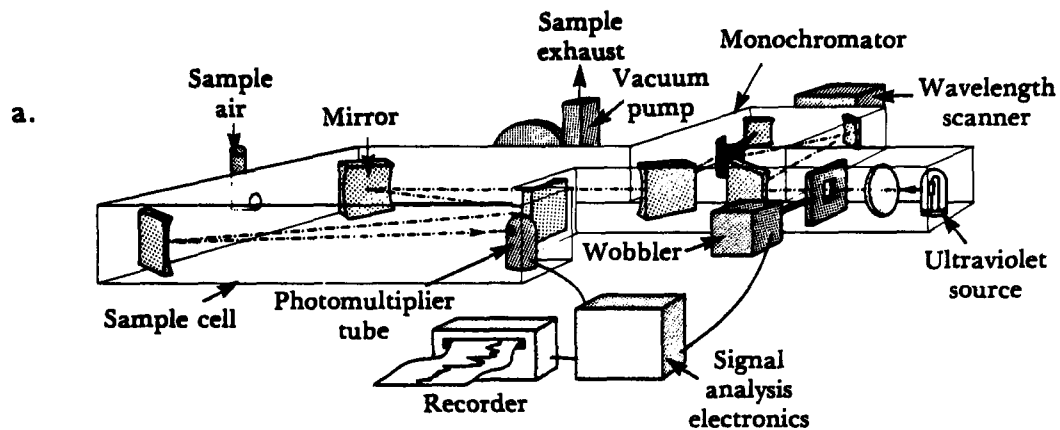For each of questions 7 through 10, match the analyzer type with its advantage(s).

7. flame photometric
8. conductimetric/amperometric
9. second-derivative spectroscopic
10. fluorescence

a. highly specific for pollutant monitored; no support gases are needed for its operation
b. no support gases are needed for its operation
c. no support gases are needed for its operation; relatively insensitive to temperature and sample-air flow variations
d. highly specific for sulfur compounds; no chemical solutions are needed for its operation; low maintenance requirements; high sensitivity for sulfur compounds; fast response
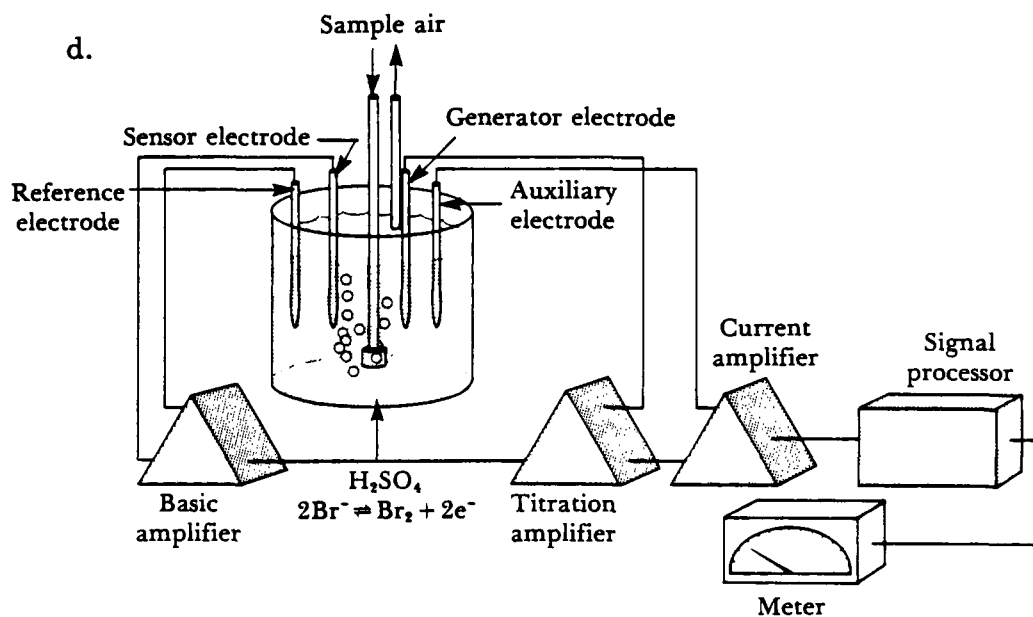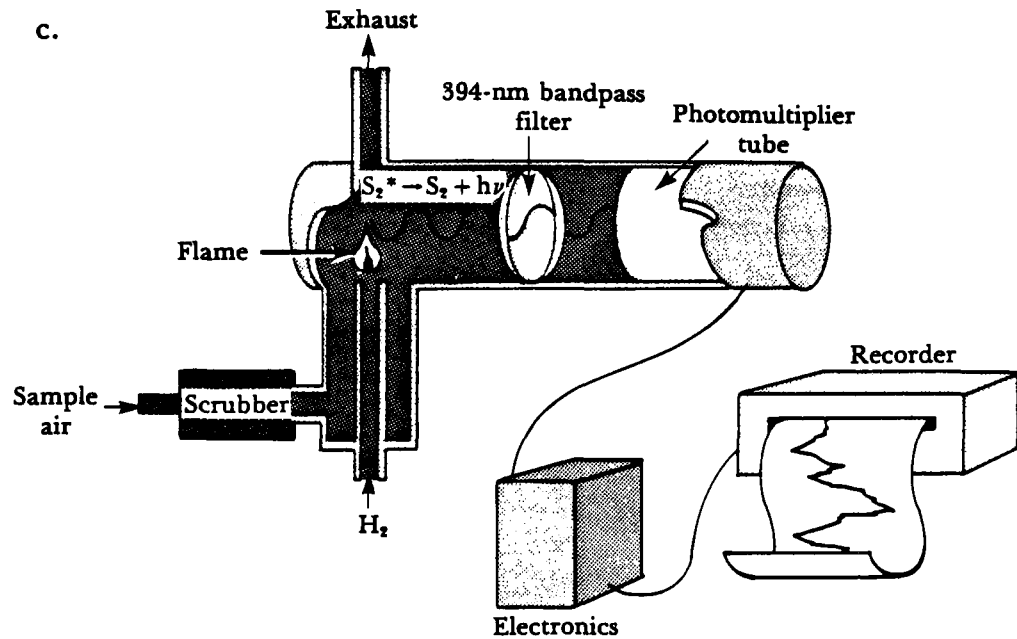
For each of questions 11 through 15, match the analyzer type with its disadvantage(s).

11. fluorescence
12. second-derivative spectroscopic
13. flame photometric
14. amperometric
15. conductimetric

a. must use a scrubber to remove sample-air components that react with halogens
b. complexity of measurement principle
c. if monitoring for SO_2, must use a scrubber to remove other sulfur compounds from the sample air; potential carbon dioxide interference; must use hydrogen
d. must use a scrubber to remove hydrocarbons and water vapor from the sample air
e. must use a peroxide solution that must be replenished frequently; inherent cycling procedure; variability of response due to interferences

For each of questions 16 through 19, match the analyzer type with its appropriate diagram.

16. amperometric
17. flame photometric
18. fluorescence
19. second-derivative spectroscopic

a.

Sample exhaust

Monochromator

Sample air

Vacuum pump

Wavelength scanner

Mirror

Wobbler

Ultraviolet source

Sample cell

Photomultiplier tube

Recorder

Signal analysis electronics

b.

210-nm bandpass filter

Exhaust

UV lamp

Sample air

350-nm bandpass filter

Photomultiplier tube

Electronics

Recorder

c.

Exhaust

394-nm bandpass filter

Photomultiplier tube

$S_2{}^* \rightarrow S_2 + h\nu$

Flame

Recorder

Sample air

Scrubber

H_2

Electronics

d.

Sample air

Sensor electrode

Generator electrode

Reference electrode

Auxiliary electrode

Current amplifier

Signal processor

Basic amplifier

H_2SO_4
$2Br^- \rightleftharpoons Br_2 + 2e^-$

Titration amplifier

Meter

For each of questions 20 through 23, match the SO_2 analyzer type with its potential major interferences.

20. amperometric
21. flame photometric
22. fluorescence

a. sulfur compounds
b. sulfur compounds, CO_2, and phosphorous
c. water vapor, oxygen, and aromatic hydrocarbons

For each of questions 23 through 28, match the SO_2 analyzer interference with an appropriate method of eliminating the interference.

23. sulfur compounds
24. CO_2
25. phosphorous
26. water vapor
27. oxygen
28. aromatic hydrocarbons

a. pass sample air through an appropriate scrubber before SO_2 is measured
b. pass sample air through a permeation dryer before SO_2 is measured
c. in calibrating the analyzer, obtain dilution air from the analyzer's sampling manifold; remove sulfur compounds from dilution air before calibrating the analyzer

29. Which of the following can be used for calibrating SO_2 analyzers?
    a. UV photometer
    b. permeation tube system
    c. standard gas cylinder/dilution system
    d. a and b, above
    e. b and c, above

30. True or False? At least two different concentrations of SO_2 should be used for calibrating an SO_2 analyzer.

31. Calibration gas should be introduced through the _____(?)_____ of an automated air analyzer.
    a. span port
    b. zero port
    c. sample handling system (probe)
    d. both a and b, above

32. True or False? It is prudent to conduct frequent zero and single-point calibration checks of automated air analyzers.

33. True or False? CO_2 tends to increase the response of flame photometric analyzers to SO_2.

34. True or False? Quenching occurs when an excited molecule loses its excess energy by colliding with another molecule.

35. Which of the following is a(are) component(s) of a clean-air package for generating zero air for calibrating flame photometric SO_2 analyzers?
    a. activated-charcoal filters
    b. molecular sieve
    c. both a and b, above
    d. none of the above

36. True or False? Burner assemblies of flame photometric SO_2 analyzers must be cleaned periodically.

37. True or False? Sample air particulate filters and scrubbers must be replaced or cleaned periodically.

38. True or False? Electrolyte solutions in amperometric SO$_2$ analyzers do not have to be replaced.

39. Which of the following is a(are) possible action(s) for eliminating a low or "noisy" response from a flame photometric SO$_2$ analyzer?
    a. clean the analyzer's burner assembly
    b. replace the analyzer's photomultiplier tube (pmt)
    c. replace the analyzer's optical window
    d. all of the above

40. True or False? Analyzer sample inlet lines, limiting orifices, flow restricting capillaries, and rotameters should be checked often.

41. True or False? Hydrogen lines, regulators, and solenoid valves do not have to be leak checked often.

42. Which of the following contain(s) quality assurance information for automated ambient air quality analyzers?
    a. *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II*
    b. *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III*
    c. *Methods for Air Sampling and Analysis*
    d. all of the above

Section 2
Review Exercise Answers

Page(s) of *Analytical Methods
for Air Quality Standards:
Student Manual*

1. c 3-1
2. b 3-1, 3-2
3. d 3-6
4. c..3-17
5. a 3-19
6. e 3-12
7. d 3-9
8. b 3-4, 3-19
9. a 3-18
10. c 3-16
11. d 3-16
12. b 3-19
13. c.................................3-7, 3-10
14. a 3-5
15. e 3-19
16. d 3-3
17. c 3-9
18. b 3-15
19. a 3-18
20. a 3-5
21. b.................................3-7, 3-10
22. c 3-16
23. a 3-5, 3-7
24. c 3-11
25. a 3-10
26. b 3-14
27. c 3-16
28. a 3-16
29. e 3-4
30. False 3-4
31. c 3-4
32. True 3-4
33. False 3-10
34. True 3-12
35. a 3-11
36. True 3-11

Section 3

Reference Methods for Ozone

Reading Assignment

Pages 4-1 through 4-17 of EPA 450/2-81-018b *APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual.*

Reading Assignment Topics

- Reference measurement principle
- Description of a typical ozone monitor
- Ultraviolet photometric calibration of ozone monitors
- Maintenance and troubleshooting of ozone monitors and calibration equipment
- Quality assurance for ozone monitors

Learning Goal and Objectives

Learning Goal

The purpose of this section is to familiarize you with reference methods for monitoring ozone.

Learning Objectives

At the end of this section, you should be able to —
1. describe the ozone reference measurement principle,
2. describe a typical chemiluminescence ozone analyzer,
3. convert between ozone concentrations expressed in parts per million (ppm) and those expressed in micrograms per standard cubic meter ($\mu g/\text{std m}^3$),
4. describe the measurement principle used by ultraviolet (UV) photometers,
5. describe an ozone generator and a typical UV photometer,
6. define and recognize two advantages of ozone transfer standards,
7. recognize two precautions for the use of ethylene,
8. identify a potential interference associated with the use of chemiluminescence ozone analyzers,
9. describe maintenance procedures for chemiluminescence ozone analyzers,
10. troubleshoot air quality monitors by using strip chart recorder traces,
11. recognize at least four situations that require an analyzer to be recalibrated, and
12. use zero and span data to determine the need for an analyzer recalibration and for data validation.

Reading Guidance

- Refer often to the figures of the assigned reading material as you progress through the assignment.
- When you have finished the reading assignment, complete the review exercise for Section 3. It begins on the following page.
- After you have answered the review exercise questions, check your answers. The correct answers are listed on the page immediately following the review exercise.
- For any review exercise questions that you answered incorrectly, review the page(s) of the reading assignment indicated on the answers page.
- After you have reviewed your incorrect answers (if any), take **Quiz 1**. Follow the directions listed in the Course Introduction section of this guidebook.
- After completing **Quiz 1**, proceed to Section 4 of this guidebook.

Review Exercise

Now that you've completed the assignment for Section 3, please answer the following questions. These will help you determine whether you are mastering the material.

1. The ozone reference measurement principle is ____(?)____.
   a. ultraviolet photometry
   b. gas-phase chemiluminescence
   c. nondispersive infrared spectroscopy
   d. flame photometry

2. The reference calibration procedure for ozone analyzers employs ____(?)____.
   a. ultraviolet photometry
   b. gas-phase chemiluminescence
   c. nondispersive infrared spectroscopy
   d. flame photometry

3. ____(?)____ ozone analyzers use the light emitted because of the reaction of ozone with ____(?)____ to measure ozone concentration.
   a. Ultraviolet photometric, ethylene
   b. Ultraviolet photometric, hydrogen
   c. Chemiluminescent, ethylene
   d. Chemiluminescent, hydrogen

4. Which of the following measurement principles is(are) used by analyzers that have been designated equivalent measurement methods for ozone?
   a. ultraviolet photometry
   b. gas-solid chemiluminescence
   c. both a and b, above
   d. gas-phase chemiluminescence
   e. a, b, and d, above

5. Which of the following is a(are) component(s) of a gas-phase chemiluminescent ozone analyzer?
   a. ultraviolet lamp
   b. reaction chamber
   c. photomultiplier tube
   d. both a and c, above
   e. both b and c, above

6. In a gas-phase chemiluminescent ozone analyzer, sample air and ethylene are mixed in a(n) ____(?)____.
   a. ozone generator
   b. vacuum pump
   c. photomultiplier tube
   d. reaction chamber

7. True or False? In a gas-phase chemiluminescent ozone analyzer, the light energy emitted by the ozone-ethylene reaction is converted to an electrical signal by a photomultiplier tube.

8. In a gas-phase chemiluminescent ozone analyzer, as the ozone concentration of the sample air ____(?)____, the light energy emitted by the ozone-ethylene reaction ____(?)____.
   a. decreases, increases
   b. increases, decreases
   c. increases, increases

9. True or False? Sample-air and ethylene flow rates must be closely controlled when using gas-phase chemiluminescent ozone analyzers.

10. True or False? Too low an ethylene flow rate could result in an erroneously high ozone-concentration reading from a chemiluminescent ozone analyzer.

11. True or False? If ethylene flow through a chemiluminescent ozone analyzer is too high, an explosion could occur.

12. At 25°C and 760 mm Hg, 0.100 ppm of ozone (molecular weight: 48 $\mu g/\mu mole$) is equal to ____(?)____ $\mu g/std\ m^3$ of ozone.
    a. 196
    b. 51
    c. 0.196
    d. 0.051

13. Which of the following can be used to prepare ozone calibration gas?
    a. compressed gas cylinders containing known amounts of ozone
    b. ozone permeation tubes
    c. ultraviolet-light sources
    d. all of the above

14. True or False? An ultraviolet photometer determines ozone concentration by measuring the absorption of ultraviolet light.

15. As ultraviolet light of an appropriate wavelength passes through an air sample containing ozone, the intensity of the light beam decreases as the ____(?)____ increases.
    a. ozone concentration of the air sample
    b. optical-path length
    c. both a and b, above
    d. none of the above

16. Ozone can be produced by irradiating with ultraviolet light the _____(?)_____ present in ozone-free air.
    a. carbon dioxide
    b. water vapor
    c. oxygen
    d. none of the above

17. Which of the following must be kept constant in order to generate constant amounts of ozone using an ultraviolet ozone generator?
    a. intensity of ultraviolet light
    b. flow rate of ozone-free air
    c. oxygen concentration of ozone-free air
    d. both a and b, above
    e. all of the above

18. Which of the following is a(are) component(s) of a UV photometer?
    a. low-pressure mercury discharge lamp
    b. absorption cell(s)
    c. detector(s)
    d. all of the above

19. Which of the following is(are) used by UV photometers in measuring ozone concentrations?
    a. current-to-frequency converters
    b. counters
    c. interference cells
    d. both a and b, above
    e. both a and c, above

20. Which of the following can be used as ozone transfer calibration standards?
    a. ozone analyzers
    b. manual analytical procedures
    c. ozone generators
    d. all of the above

21. Which of the following is an(are) advantage(s) of using ozone transfer calibration standards?
    a. allows the calibrations of ozone analyzers at several monitoring sites to be referenced to one centrally located UV photometer
    b. eliminates the transporting of UV photometers to monitoring sites
    c. both a and b, above

22. True or False? Measurements of very low ozone concentrations made by gas-phase chemiluminescent analyzers can be affected by humid sample air.

23. Which of the following is(are) necessary to prevent the acccumulation of explosive concentrations of ethylene in monitoring shelters containing gas-phase chemiluminescent ozone analyzers?
a. vent chemiluminescent ozone analyzers to the outside air
b. leak check ethylene gas handling systems
c. use an ethylene scrubber
d. both a and b, above
e. both a and c, above

24. Which of the following components of a gas-phase chemiluminescent ozone analyzer must be checked periodically?
a. Teflon® sample-air inlet filter
b. ethylene and sample-air pressures and flow rates
c. gas-handling systems (for leaks)
d. all of the above

25. True or False? The sintered filter located in the ethylene gas handling system of a gas-phase chemiluminescent ozone analyzer never has to be replaced.

For each of questions 26 through 30, match the strip-chart recorder trace with its possible causes.

26.
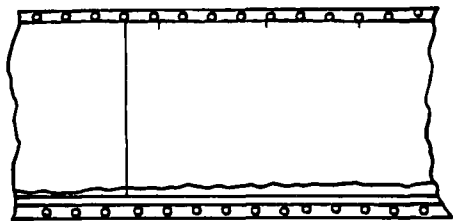
Straight line trace sustained
for several hours

a. stoppage of sample air or ethylene flow; electrical failure in detection circuit

b. unusually large decrease in shelter temperature or electrical line voltage
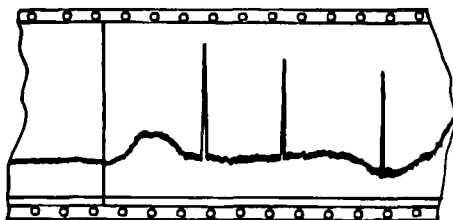
c. shelter temperature fluctuations beyond operating temperature limits of analyzer

27.
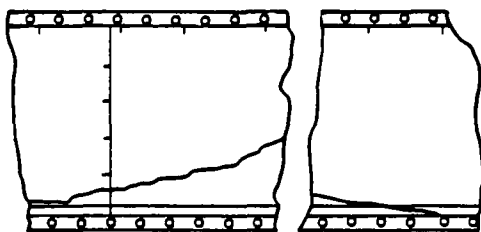
Drift, gradually increasing
or decreasing

d. electrical instability of analyzer or a dirty photomultiplier tube window
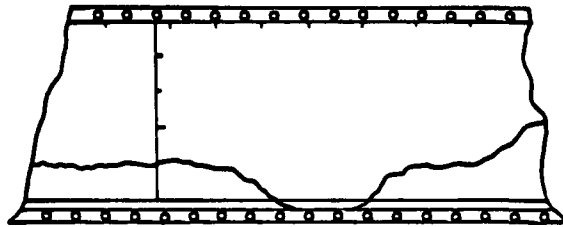
e. a leak in or gradual clogging of analyzer's gas-handling system; failure of detector-cell temperature controller

28.

Wide solid trace, or with
sharp "spikes"

29.
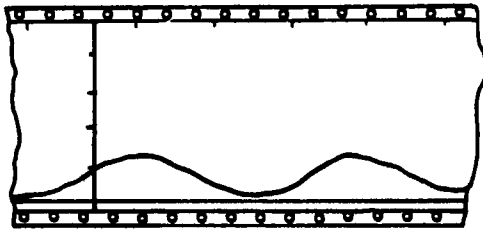
Cyclic pattern with
definite time period

30.

Recorded trace drops below
baseline for periods of time,
possibly returning

31. In which of the following situations should an air-quality analyzer be recalibrated?
   a. after any repair that may affect the analyzer's calibration
   b. following the physical relocation of the analyzer
   c. when there is any indication of possible significant inaccuracy of the analyzer
   d. both a and c, above
   e. a, b, and c, above

32. True or False? Analyzer zero and span drift limits can be used to determine the need for analyzer recalibration.

33. True or False? Analyzer zero and span drift limits cannot be used for data validation.

34. True or False? Visual evaluation of strip chart recorder traces can be used for data validation.

Section 3
Review Exercise Answers

Section 4

Reference Method for Total Suspended Particulate Matter (TSP)

Reading Assignment

Pages 5-1 through 5-29 of EPA 450/2-81-018b *APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual.*

Reading Assignment Topics

- Description of the high volume sampler
- Filter handling
- Calibration of the high volume sampler
- Sampling and analysis procedure for TSP
- Factors affecting high volume sampling accuracy and precision
- Quality assurance considerations

Learning Goal and Objectives

Learning Goal

The purpose of this section is to familiarize you with the reference method for measuring total suspended particulate matter.

Learning Objectives

At the end of this section, you should be able to—
1. recognize the use of a high volume sampler in the TSP reference method,
2. recognize at least three major components of a high volume sampler,
3. identify and give the locations of at least five components of a calibration set-up for calibrating a high volume sampler orifice-type flow rate transfer standard,
4. recognize that a flow rate transfer standard is needed to calibrate a high volume sampler's flow rate measuring device,
5. compare a calibration curve for a high volume sampler flow rate transfer standard to a high volume sampler flow rate measuring device calibration curve,
6. identify and describe how to minimize potential sources of error in high volume sampling,
7. recognize the use of Reference Flow (ReF) devices for auditing flow rate calibrations of high volume samplers,

8. identify at least four components of a high volume sampler that need periodic cleaning or replacement,

9. recognize at least four reasons to use a shelter when a high volume sampler is used to sample suspended particulate matter,

10. calculate total suspended particulate matter concentration from high volume sampling and analysis data,

11. recognize at least three advantages of glass-fiber filters and at least two disadvantages of cellulose filters,

12. describe in sequence the activities involved in the reference method for TSP, and

13. recognize at least seven items that should be checked during performance and systems audits.

Reading Guidance

- Refer often to the equations and figures of the assigned reading material as you progress through the assignment.
- When you have finished the reading assignment, complete the review exercise for Section 4. It begins on the following page.
- After you have answered the review exercise questions, check your answers. The correct answers are listed on the page immediately following the review exercise.
- For any review exercise questions that you answered incorrectly, review the page(s) of the reading assignment indicated on the answers page.
- After you have reviewed your incorrect answers (if any), proceed to Section 5 of this guidebook.

Review Exercise

Now that you've completed the assignment for Section 4, please answer the following questions. These will help you determine whether you are mastering the material.

1. Which of the following is a(are) major component(s) of a high volume sampler?
   a. modified vacuum-sweeper motor
   b. stainless steel filter holder
   c. photodetector
   d. both a and b, above
   e. both a and c, above

2. Which of the following is a(are) reason(s) for the use of a shelter when sampling suspended particulate matter using a high volume sampler?
   a. Shelter protects filter from direct impingement of particulate matter.
   b. Shelter uniformly distributes particulate matter over the filter surface.
   c. Shelter prevents birds from damaging filter.
   d. both a and b, above
   e. both b and c, above

3. True or False? When using a high volume sampler, the sampling flow rate affects the size of particles collected.

4. Which of the following is *not* a reason for the use of glass-fiber filters in routine high volume sampling?
   a. They have collection efficiencies of at least 99%.
   b. They have low resistance to air flow.
   c. They have low affinity for moisture.
   d. They are suitable for the analysis of many organic and inorganic particulate pollutants.
   e. They cost less than do other filters.

5. In order to eliminate weight errors due to small amounts of moisture, both unexposed and exposed glass-fiber filters should be equilibrated at _____(?)_____ °C and less than _____(?)_____ percent relative humidity for 24 hours before weighing.
   a. 0 to 5, 50
   b. 15 to 35, 25
   c. 15 to 30, 50
   d. 15 to 35, 75

6. True or False? Blank concentrations of pollutants in clean glass-fiber filters should be taken into account when analyzing exposed filters for pollutants.

7. Which of the following is a(are) disadvantage(s) of using cellulose filters for high volume sampling?
   a. By rapidly clogging, they cause sampling flow rates to dramatically decrease.
   b. They have low metal content.
   c. They enhance the artifact formation of sulfates and nitrates.
   d. both a and c, above
   e. both b and c, above

8. True or False? Folding or creasing a high volume filter before sampling may cause erroneous flow patterns through the filter during sampling.

9. True or False? If an exposed high volume filter's border is fuzzy or nonexistent, sample air may have leaked around the filter's gasket during sampling.

10. True or False? For transport to the laboratory for analysis after sampling, an exposed high volume filter should be folded in half lengthwise so that collected particulate matter on one half of the filter does not touch collected particulate matter on the other half of the filter.

11. Which of the following are devices used to measure sampling flow rates of high volume samplers?
    a. orifice/pressure indicators
    b. mass flowmeters
    c. Roots meters
    d. a and b, above
    e. a, b, and c, above

12. In the calibration set-up for the high volume sampler flow rate transfer standard depicted below, a, b, c, d, and e are the ____(?)____ respectively.
    a. water manometer, mercury manometer, flow rate transfer standard, Roots meter, and high volume motor
    b. mercury manometer, water manometer, flow rate transfer standard, Roots meter, and high volume motor
    c. mercury manometer, water manometer, high volume motor, Roots meter, and flow rate transfer standard
    d. water manometer, mercury manometer, flow rate transfer standard, high volume motor, and Roots meter
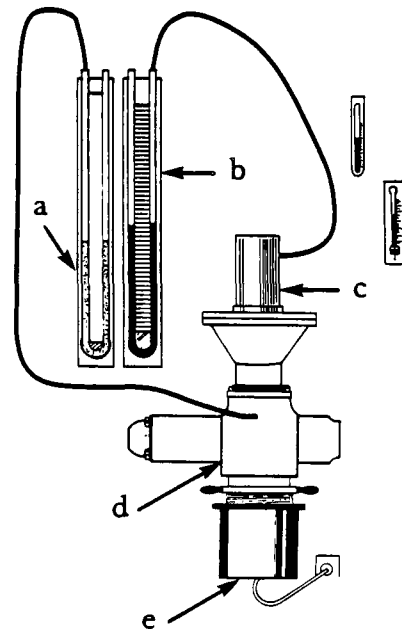
13. The U.S. EPA high volume sampling procedure requires that high volume samplers be operated at sampling flow rates of ____(?)____ to ____(?)____ m³/min.
    a. 1.0, 2.0
    b. 1.1, 1.7
    c. 1.0, 1.5

14. A calibration curve for a high volume sampler flow rate transfer standard is constructed by plotting ____(?)____ versus ____(?)____.
    a. standard flow rate, $\sqrt{I(P_2/P_{std})(298/T_2)}$
    b. flow rate transfer standard pressure drop, Roots meter pressure drop
    c. standard flow rate, $\sqrt{\Delta H(P_1/P_{std})(298/T_1)}$
    d. standard flow rate, indicated flow rate

15. Which of the following items is(are) needed to calibrate a high volume sampler's flow rate measuring device?
    a. rotameter
    b. elutriator
    c. mass flow controller
    d. flow rate transfer standard
    e. both a and b, above
    f. all of the above

16. Which of the following equations is used to correct air volumes measured by positive-displacement standard volume meters to standard air volumes for the calibration of high volume sampler flow rate transfer standards?

    a. $V_{std} = V_m \left(\dfrac{P_1}{P_{std} - \Delta P} \right) \left(\dfrac{T_{std}}{T_1} \right)$,

    b. $V_{std} = V_m \left(\dfrac{P_1 - \Delta P}{P_{std} - \Delta P} \right) \left(\dfrac{T_{std}}{T_1} \right)$

    c. $V_{std} = V_m \left(\dfrac{P_1 - \Delta P}{P_{std}} \right) \left(\dfrac{T_1}{T_{std}} \right)$

    d. $V_{std} = V_m \left(\dfrac{P_1 - \Delta P}{P_{std}} \right) \left(\dfrac{T_{std}}{T_1} \right)$

    Where: V_{std} = standard volume, std m³
              V_m = actual volume measured by the standard volume meter
              P_1 = barometric pressure during calibration, mm Hg or kPa
              ΔP = pressure drop at inlet to volume meter, mm Hg or kPa
              P_{std} = 760 mm Hg or 101 kPa
              T_{std} = 298 K
              T_1 = ambient temperature during calibration, K

17. A calibration curve for a high volume sampler is constructed by plotting
    ____(?)____ versus ____(?)____ .
    a. standard flow rates, appropriately expressed flow rates indicated by the high volume sampler's flow rate measuring device
    b. flow rate transfer standard pressure drops, appropriately expressed flow rates indicated by the high volume sampler's flow rate measuring device
    c. standard flow rates, flow rate transfer standard pressure drops

18. Which of the following is a(are) potential source(s) of error in high volume sampling?
    a. nonuniform flow rate changes during sampling
    b. wind directional sensitivity caused by the gabled roof of the high volume sampler's shelter
    c. artifact particulate matter formation on alkaline high volume filters
    d. all of the above

19. Which of the following devices can minimize errors caused by nonuniform changes in high volume sampler flow rates during sampling?
    a. pressure transducer with continuous recorder for flow rate
    b. constant flow rate controllers
    c. both a and b, above
    d. none of the above

20. True or False? A Reference Flow (ReF) device can be used to audit the flow rate calibrations of high volume samplers.

21. True or False? Mass flow controllers maintain a constant high volume sampler flow rate by adjusting the speed of the high volume sampler's motor during sampling.

22. True or False? Slightly increasing the line voltage to a high volume sampler's motor will increase motor brush life.

23. Which of the following components of a high volume sampler does *not* need periodic cleaning or replacement?
    a. flow rate measuring device
    b. tubing used in flow-rate indication
    c. faceplate gasket
    d. motor gaskets
    e. none of the above

24. Under the conditions described below, the suspended particulate concentration obtained using a high volume sampler is ____(?)____ $\mu g/std\ m^3$.
    a. 107
    b. 94
    c. 100   Given: Weight of filter after sampling: 3.216 g
    d. 6   Weight of filter before sampling: 3.000 g
                       Initial sampling flow rate: 1.60 std m^3/min
                       Final sampling flow rate: 1.40 std m^3/min
                       Sampling period: midnight 7-11-81 to midnight 7-12-81

25. True or False? A high volume sampler is used in the reference method for total suspended particulate matter (TSP).

26. True or False? The shelter of a high volume sampler prevents particles that are greater than 5 μm in diameter from being collected by the sampler.

27. True or False? Motor brushes of a high volume sampler must be periodically replaced.

28. A high volume sampler should be recalibrated after which of the following?
    a. replacement of the particulate matter filter
    b. replacement of the sampler's motor
    c. replacement of the sampler motor's brushes
    d. both a and b, above
    e. both b and c, above

29. Which of the following is the proper sequence of activities for preparing a high volume particulate filter for sampling?
    a. visually inspect the filter, number the filter, equilibrate the filter, weigh the filter
    b. number the filter, visually inspect the filter, weigh the filter, equilibrate the filter
    c. number the filter, visually inspect the filter, equilibrate the filter, weigh the filter
    d. number the filter, equilibrate the filter, weigh the filter, visually inspect the filter

30. High volume particulate filters must be weighed to the nearest ____(?)____ mg.
    a. 1
    b. 10
    c. 0.1

31. True or False? Filter cartridges can be used to eliminate the handling of high volume particulate filters at sampling sites.

32. Which of the following sampling data must be recorded at the total suspended particulate matter (TSP) sampling site?
    a. identification of particulate-matter filter and sampling site
    b. initial and final sampler flow rates
    c. initial weight of the particulate filter
    d. both a and b, above
    e. all of the above

33. True or False? Exposed particulate filters must be re-equilibrated before they are weighed.

34. A high volume particulate-matter filter can be analyzed for which of the following?
    a. organic compounds
    b. trace metals
    c. all of the above

35. True or False? A variable resistance orifice does not require a set of resistance plates for the calibration of high volume sampler flow rates.

36. True or False? Before a flow rate transfer standard is calibrated, its calibration set-up must be checked for air leaks.

37. True or False? The precision of high volume sampling data can be determined by using collocated high volume samplers.

38. Performance audits should be conducted for which of the following activities of the reference method for total suspended particulate matter?
    a. filter weighing
    b. sampler flow rate calibration
    c. data processing
    d. a and b, above
    e. a, b, and c, above

39. Which of the following should be determined during a systems audit of a total suspended particulate matter (TSP) monitoring network?
    a. types of high volume samplers and particulate-matter filters used
    b. sampling, analysis, data processing, and calibration procedures used
    c. whether there is sufficient documentation of TSP monitoring activities
    d. b and c, above
    e. a, b, and c, above

Section 4
Review Exercise Answers

Section 5

Reference Methods
for Carbon Monoxide

Reading Assignment

Pages 6-1 through 6-15 of EPA 450/2-81-018b, *APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual.*

Reading Assignment Topics

- Reference measurement principle
- Description of a Luft-type carbon monoxide analyzer
- Calibration of carbon monoxide analyzers
- Interferences
- Description of a gas-filter correlation carbon monoxide analyzer
- Maintenance and troubleshooting of carbon monoxide analyzers
- Quality assurance for carbon monoxide analyzers

Learning Goal and Objectives

Learning Goal

The purpose of this section is to familiarize you with reference methods for monitoring carbon monoxide.

Learning Objectives

At the end of this section, you should be able to—
1. describe the carbon monoxide reference measurement principle,
2. describe typical nondispersive infrared (NDIR) carbon monoxide analyzers,
3. describe the calibration of carbon monoxide analyzers,
4. identify at least two potential interferences associated with NDIR carbon monoxide analyzers and at least three methods for their removal,
5. describe maintenance and troubleshooting procedures for NDIR carbon monoxide analyzers,
6. recognize at least three parameters associated with carbon monoxide analyzers that require quality control, and
7. describe a water vapor interference check for carbon monoxide analyzers.

Reading Guidance

- Refer often to the tables and figures of the assigned reading material as you progress through the assignment.
- When you have finished the reading assignment, complete the review exercise for Section 5. It begins on the following page.
- After you have answered the review exercise questions, check your answers. The correct answers are listed on the page immediately following the review exercise.
- For any review exercise questions that you answered incorrectly, review the page(s) of the reading assignment indicated on the answers page.
- After you have reviewed your incorrect answers (if any), proceed to Section 6 of this guidebook.

Review Exercise

Now that you've completed the assignment for Section 5, please answer the following questions. These will help you determine whether you are mastering the material.

1. The carbon monoxide reference measurement principle uses ____(?)____.
   a. absorption of ultraviolet radiation
   b. absorption of infrared radiation
   c. flame photometry
   d. gas-phase chemiluminescence

2. True or False? Carbon monoxide analyzers use diffraction gratings to isolate a specific infrared wavelength for absorption by carbon monoxide.

For each of questions 3 through 9, match the analyzer component with its appropriate figure in the diagram of a typical Luft-type nondispersive infrared (NDIR) carbon monoxide analyzer shown below.

   3. infrared source
   4. beam chopper
   5. bandpass filter
   6. sample cell
   7. detector
   8. reference cell
   9. diaphragm
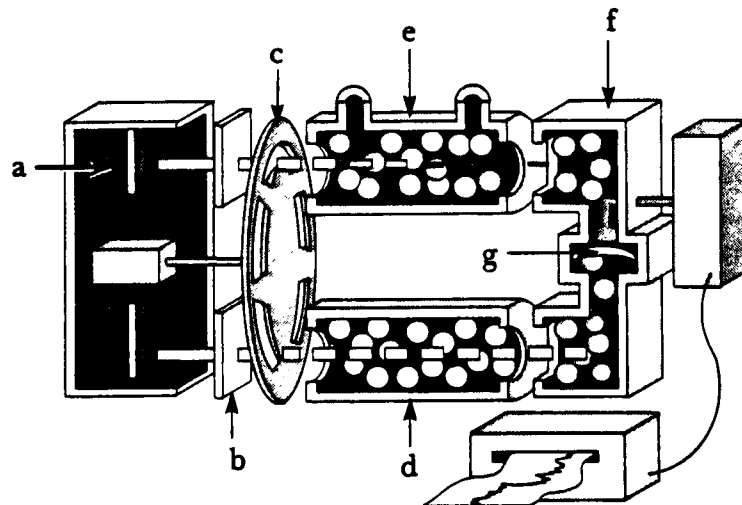
10. A(n) _____(?)_____ is usually used as the source of infrared radiation in an NDIR carbon monoxide analyzer.
    a. infrared lamp
    b. photomultiplier tube
    c. tungsten lamp
    d. heated wire

11. True or False? The beam chopper of an NDIR carbon monoxide analyzer helps amplify the analyzer detector's output.

12. Infrared radiation emerging from the sample cell of a Luft-type NDIR carbon monoxide analyzer should always be _____(?)_____ the infrared radiation emerging from the analyzer's reference cell.
    a. less than
    b. greater than
    c. equal to
    d. less than or equal to
    e. greater than or equal to

13. The detector cell of a Luft-type NDIR carbon monoxide analyzer consists of _____(?)_____ compartments filled with equal concentrations of _____(?)_____.
    a. 3, carbon monoxide
    b. 2, carbon monoxide
    c. 3, nitrogen
    d. 2, nitrogen

14. True or False? The detector compartments of a Luft-type NDIR carbon monoxide analyzer are separated by an immovable diaphragm.

15. True or False? When carbon monoxide is present in the sample cell of a Luft-type NDIR carbon monoxide analyzer, the analyzer's diaphragm bends into the sample compartment of the analyzer's detector.

16. True or False? In calibrating an NDIR carbon monoxide analyzer, calibration gases should be introduced through the analyzer's sampling line.

17. Which of the following is a(are) component(s) of a gas-filter correlation NDIR CO analyzer?
    a. gas cell wheel
    b. sample chamber
    c. reference cell
    d. a and b, above
    e. a, b, and c, above

18. True or False? Carbon monoxide cylinder gases should consist of carbon monoxide and dry nitrogen if they are to be diluted less than 100:1 with air when preparing carbon monoxide calibration gases.

19. True or False? The gas-filter correlation technique greatly reduces interference effects associated with NDIR CO analyzers.

20. True or False? Water vapor and carbon dioxide are potential negative interferences in the NDIR measurement of carbon monoxide.

21. Which of the following can be used to minimize *both* water vapor and carbon dioxide interferences in the NDIR measurement of carbon monoxide?
    a. silica gel
    b. refrigeration units
    c. negative filtering
    d. interference cells
    e. both a and b, above
    f. both c and d, above

22. ___(?)___ fluctuations can cause changes in the response of an NDIR carbon monoxide analyzer.
    a. Temperature
    b. Pressure
    c. Temperature and pressure
    d. none of the above

23. True or False? Sampling flow rate fluctuations can cause changes in the response of an NDIR carbon monoxide analyzer.

24. True or False? Particulate matter must be removed from sample air before the sample air enters the sample cell of an NDIR carbon monoxide analyzer.

25. True or False? All Luft-type NDIR carbon monoxide analyzers are insensitive to mechanical vibration.

26. Which of the following components of an NDIR carbon monoxide analyzer should be periodically checked?
    a. vacuum pump
    b. infrared radiation source(s)
    c. optical system
    d. b and c, above
    e. a, b, and c, above

27. Which of the following are possible causes for a Luft-type carbon monoxide analyzer to yield erroneously high carbon monoxide concentrations?
    a. reference infrared radiation source failing
    b. sample infrared radiation source failing
    c. decreased pressure in reference compartment of detector
    d. decreased pressure in sample compartment of detector
    e. both a and c, above
    f. both b and d, above

28. Which of the following are possible causes for a Luft-type carbon monoxide analyzer to yield erroneously low carbon monoxide concentrations?
    a. reference infrared radiation source failing
    b. sample infrared radiation source failing
    c. decreased pressure in reference compartment of detector
    d. decreased pressure in sample compartment of detector
    e. both a and c, above
    f. both b and d, above

29. If a water vapor interference check of an NDIR carbon monoxide analyzer results in a difference between dry and saturated values of ____(?)____ ppm or greater, the analyzer's water vapor removal system must be repaired or replaced.
    a. 0.1
    b. 0.5
    c. 1.0
    d. 2.0

30. Which of the following operating parameters of an NDIR carbon monoxide analyzer should be routinely checked?
    a. zero drift
    b. span drift
    c. sample cell pressure
    d. a and b, above
    e. a, b, and c, above

Section 5
Review Exercise Answers

Section 6

Reference Methods
for Nitrogen Dioxide

Reading Assignment

Pages 7-1 through 7-14 of EPA 450/2-81-018b *APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual.*

Reading Assignment Topics

- Reference measurement principle
- Description of typical nitrogen dioxide analyzers
- Calibration of nitrogen dioxide analyzers
- Interferences
- Maintenance and troubleshooting of nitrogen dioxide analyzers
- Quality assurance for nitrogen dioxide analyzers

Learning Goal and Objectives

Learning Goal

The purpose of this section is to familiarize you with reference methods for monitoring nitrogen dioxide.

Learning Objectives

At the end of this section, you should be able to—
1. describe the nitrogen dioxide reference measurement principle,
2. describe typical chemiluminescent nitrogen dioxide analyzers,
3. describe the gas phase titration (GPT) and permeation tube methods of calibrating nitrogen dioxide analyzers,
4. identify at least two potential interferences associated with chemiluminescent nitrogen dioxide analyzers,
5. describe maintenance and troubleshooting procedures for chemiluminescent nitrogen dioxide analyzers,
6. recognize at least four components of a nitrogen dioxide measurement system that require visual inspection, and
7. recognize two types of performance audits for nitrogen dioxide analyzers.

Reading Guidance

- Refer often to the table and figures of the assigned reading material as you progress through the assignment.
- When you have finished the reading assignment, complete the review exercise for Section 6. It begins on the following page.
- After you have answered the review exercise questions, check your answers. The correct answers are listed on the page immediately following the review exercise.
- For any review exercise questions that you answered incorrectly, review the page(s) of the reading assignment indicated on the answers page.
- After you have reviewed your incorrect answers (if any), take **Quiz 2.** Follow the directions listed in the Course Introduction section of this guidebook.
- After completing **Quiz 2,** proceed to Section 7 of this guidebook.

Review Exercise

Now that you've completed the assignment for Section 6, please answer the following questions. These will help you determine whether you are mastering the material.

1. The reference measurement principle for nitrogen dioxide (NO_2) employs _____(?)_____.
   a. gas-phase chemiluminescence
   b. flame photometry
   c. nondispersive infrared spectroscopy
   d. ultraviolet photometry

2. In the NO_2 reference measurement principle, light is emitted as a result of _____(?)_____ reacting with _____(?)_____.
   a NO_2, ozone (O_3)
   b. NO, ozone
   c. NO_2, NO
   d. NO, oxygen (O_2)

3. True or False? NO_2 is measured directly by NO_2 analyzers.

4. True or False? NO_2 must be converted to NO before it can be measured by NO_2 analyzers.

5. Which of the following equations describes the chemiluminescent reaction of the NO_2 reference measurement principle?
   a. $NO_2 + O_3 \rightarrow NO^* + 2O_2$
      $NO^* \rightarrow NO + h\nu$
   b. $NO + O_3 \rightarrow NO_2{}^* + O_2$
      $NO_2{}^* \rightarrow NO_2 + h\nu$
   c. $NO_2 + NO \rightarrow N_2 + O_3{}^*$
      $O_3{}^* \rightarrow O_3 + h\nu$
   d. $NO + 2O_2 \rightarrow NO_2{}^* + O_3$
      $NO_2{}^* \rightarrow NO_2 + h\nu$

6. Cyclic chemiluminescent NO_2 analyzers have _____(?)_____ reaction chamber(s) and _____(?)_____ detector(s).
   a. 1, 1
   b. 1, 2
   c. 2, 1

7. True or False? In a dual chamber chemiluminescent NO_2 analyzer, sample air flows continuously through a converter.

8. True or False? Varying concentrations of ozone are supplied to the reaction chamber of a chemiluminescent NO_2 analyzer by an internal ozone generator.

9. The reaction chamber of a chemiluminescent NO_2 analyzer is maintained ____(?)____ atmospheric pressure.
   a. at
   b. below
   c. above

10. An optical filter is placed between the reaction chamber and photomultiplier tube of a chemiluminescent NO_2 analyzer to eliminate interfering emissions caused by the reaction of ozone with ____(?)____.
    a. NO_2
    b. NO
    c. NO_2*
    d. unsaturated hydrocarbons

11. True or False? Heating the photomultiplier tube (PMT) of a chemiluminescent NO_2 analyzer increases its sensitivity.

12. Which of the following is a(are) method(s) allowed by U.S. EPA for the calibration of an NO_2 analyzer?
    a. gas phase titration
    b. NO_2 permeation tube/dilution system
    c. ultraviolet photometry
    d. both a and b, above
    e. both b and c, above

13. Which of the following must be calibrated during the calibration of an NO_2 analyzer?
    a. analyzer's NO_2 response
    b. analyzer's NO response
    c. analyzer's NO_x response
    d. a and b, above
    e. a, b, and c, above

14. In the gas phase titration calibration of an NO_2 analyzer, ____(?)____ is added to excess ____(?)____ in a dynamic calibration system to generate known concentrations of NO_2.
    a. NO, ozone
    b. ozone, NO
    c. NO, NO_2
    d. NO_2, NO

15. A gas phase titration system that is used to calibrate an NO_2 analyzer must have a dynamic parameter specification equal to or greater than ____(?)____ ppm-minutes.
    a. 1.00
    b. 2.00
    c. 2.75
    d. 3.75

16. Cylinder gas used in the calibration of an NO_2 analyzer must consist of NO and _____(?)_____ having an NO_2 impurity of less than _____(?)_____ ppm.
    a. nitrogen, 2
    b. air, 2
    c. oxygen, 1
    d. nitrogen, 1

17. True or False? In a gas-phase titration calibration system for an NO_2 analyzer, the mixing chamber must be downstream of the reaction chamber.

18. True or False? Flushing a gas-phase titration system with NO before it is used to calibrate an NO_2 analyzer assures that there is no NO_2 present at the beginning of the calibration.

19. True or False? In a gas-phase titration calibration of an NO_2 analyzer, the analyzer's NO response must be calibrated before its NO_2 response.

20. An NO_2 analyzer must have an average converter efficiency of _____(?)_____ percent or greater.
    a. 26
    b. 56
    c. 76
    d. 96

21. True or False? An NO_2 permeation tube consists of a tube that is partially filled with liquefied NO_2. The gaseous NO_2 above the liquid is able to permeate the walls of the tube.

22. True or False? Purge gas passing over a permeation tube in an NO_2 permeation tube/dilution system used to calibrate an NO_2 analyzer does not have to be maintained at a constant temperature.

23. True or False? If an NO_2 analyzer is calibrated by using an NO_2 permeation tube/dilution system, its converter efficiency does not have to be determined.

24. Which of the following is a(are) potential interference(s) in the gas-phase chemiluminescent measurement of NO_2?
    a. PAN
    b. organic nitrates
    c. organic nitrites
    d. a and b, above
    e. a, b, and c, above

25. True or False? Chemical converters of NO_2 analyzers never have to be replaced or reactivated.

26. Ammonia is oxidized to NO by NO_2 analyzer thermal converters operating at temperatures of _____(?)_____ °C or greater.
    a. 100
    b. 250
    c. 350
    d. 600

27. An NO_2 analyzer that has an ineffective NO_2 converter will yield erroneously low ____(?)____ concentrations.
    a. NO
    b. NO_x
    c. NO_2
    d. both a and b, above
    e. both b and c, above

28. Which of the following operating parameters of an NO_2 monitoring system should be routinely checked?
    a. temperature of monitoring shelter
    b. sample-air introduction system
    c. operation of NO_2 analyzer/recording system
    d. b and c, above
    e. a, b, and c, above

29. Performance audits should be conducted for which of the following activities of the gas-phase chemiluminescent measurement of NO_2?
    a. NO_2 analyzer multipoint calibration
    b. NO_2 data reduction
    c. both a and b, above

Section 6
Review Exercise Answers

Section 7

Reference Method for Lead

Reading Assignment

Pages 7-3 through 7-42 of this guidebook.

Reading Assignment Topics

- General description of the reference method
- Sample analysis
- Calculations and data reporting
- Maintenance and troubleshooting
- Auditing

Learning Goal and Objectives

Learning Goal

The purpose of this section is to familiarize you with the reference method for measuring lead.

Learning Objectives

At the end of this section, you should be able to—
1. recognize the use of a high volume sampler in the lead reference method,
2. describe minimum specifications for equipment and reagents used in the analysis of lead,
3. identify two sample extraction procedures and recognize critical steps in sample extraction,
4. recognize at least two important considerations for sample analysis using an atomic absorption spectrophotometer,
5. describe the calibration of an atomic absorption spectrophotometer,
6. calculate lead concentration from sampling and analysis data,
7. describe maintenance and troubleshooting procedures for an atomic absorption spectrophotometer,
8. describe performance audits for high volume sampler flow rate, lead analysis, and data processing, and
9. recognize at least three items that should be checked during a system audit for the lead reference method.

Reading Guidance

- Because the lead reference method uses the high volume sampler method (described in Section 4 of this guidebook) for sample collection, this section deals mainly with lead analysis.
- The first word in the 13th line of Section 6.1 of the reading assignment should be "more" not "less."
- Refer often to the equations, tables, and figures of the assigned reading material as you progress through the assignment.
- When you have finished the reading assignment, complete the review exercise for Section 7. It begins on page 7-43.
- After you have answered the review exercise questions, check your answers. The correct answers are listed on the page immediately following the review exercise.
- For any review exercise questions that you answered incorrectly, review the page(s) of the reading assignment indicated on the answers page.
- After you have reviewed your incorrect answers (if any), proceed to Section 8 of this guidebook.

Excerpts of Section 2.8 of
Quality Assurance Handbook for
Air Pollution Measurement Systems,
Volume II EPA 600/4-77-027a

Section 2.8

REFERENCE METHOD FOR THE DETERMINATION OF LEAD IN
SUSPENDED PARTICULATE MATTER COLLECTED FROM
AMBIENT AIR

SUMMARY

Ambient air is drawn through a glass fiber filter of a hi-vol sampler to collect particulate material. The lead concentration in the suspended particulate matter is analyzed by atomic absorption spectrophotometry.[1]

This method of sampling is applicable to measurement of the mass concentration of suspended particulates in ambient air. The size of the sample collected is usually adequate for other analyses. When the sampler is operated 24 h at an average flow rate of 1.70 m^3/min (60.0 ft^3/min) an adequate sample is obtained, even in an atmosphere having a concentration of suspended particulates as low as 1 $\mu g/m^3$.

The typical range of the method is 0.07 to 7.5 μg Pb/m^3 assuming an upper linear range of analysis of 15 $\mu g/ml$ and an air volume of 2400 m^3. Typical sensitivities for a 1% change in absorption (0.0044 absorbance units) are 0.2 and 0.5 μg Pb/ml for the 217.0 and 283.3 nm lines, respectively. A typical lower detectable limit (LDL) is 0.07 μg Pb/m^3 when an air volume of 2400 m^3 is assumed. The value quoted in the Federal Register[1] (0.07 μg Pb/m^3) was derived from instruments of different models using the 283.3 and the 217.0 nm Pb lines.

Absolute values for individual laboratories will vary with the type of instrument, the absorption wavelength, and the instrumental operating conditions.

The method description which follows is based on the promulgated Reference Method.[1]

2.2 Atomic Absorption Spectrophotometer

Major repairs and adjustment of the atomic absorption spectrophotometer normally require the services of the manufacturer or the manufacturer's representative. However, before operating the spectrophotometer, instrument performance, such as sensitivity and reproducibility should be checked by using a standard metal solution. The instrument sensitivity depends on various factors such as alignment of the hollow cathode lamp and burner head, cleanness of optical systems and burner head, and the level of grating system calibration. An optimal sensitivity is recommended by the manufacturer in the instrument manual. After this sensitivity is achieved, check the reproducibility. Typical sensitivities for a 1% change in absorption are 0.2 and 0.5 μg Pb/ml for wavelengths of 217.0 and 283.3 nm, respectively. An instrument reproducibility change of ±5% is acceptable; repeat the analysis until this limit is achieved. If the reproducibility varies by more than ±5%, the instrument should be checked by a manufacturer's representative or a qualified operator.

The following step-by-step reproducibility test procedure is recomended:

1. Prepare a series of standard lead solutions containing 0.2, 1.6, and 10.0 μg Pb/ml, or 3 standard concentrations that "bracket" the normal sample concentration range, as described in Section 2.8.5, Subsection 5.7.2.

2. Set the atomic absorption spectrophotometer for standard conditions, as described in Section 2.8.5, Subsection 5.6.

3. Take three or more readings for each standard metal solution prepared in step 1. This can be done either sequentially or by alternating the standards, however, calibration standards should be analyzed at random throughout the analysis to check the calibration stability. See Section 2.8.5, Subsection 5.7.3.

4. Record the instrument response in absorbance units. If using a strip chart recorder, take the mid point of the noise as baseline and measure the net difference between the baseline

noise and the peak height. If the instrument has a digital read-out and printer, there is no need to manually measure peaks and absorbance units can be read directly.

5. Determine the instrument's reproducibility by subtracting the lowest response from the highest response, then dividing by average response, and multiplying by 100. Correct these values by subtracting the blank. For example, assume that the three response values derived from a 10.0 µg Pb/ml standard solution are:

Absorbance	Peak response minus blank
Blank = 0.005	
1st peak = 0.100	0.095
2nd peak = 0.098	0.093
3rd peak = 0.098	0.093

The average response value for these three analyses is 0.094, which represents the standard metal solution's concentration (10.0 µg Pb/ml). The maximum percentage absorbance fluctuation is given by:

$$\text{Percent fluctuation} = \frac{100 \times (0.095 - 0.093)}{0.094} = 2\%.$$

The percent fluctuation should be $\leq 5\%$; if not, have the instrument checked by a qualified service engineer.

5.0 ANALYSIS OF SAMPLES

Table 5.1 at the end of this section summarizes the major quality assurance activities for sample analyses.

5.2 General Analysis Description

In atomic absorption spectrophotometry, the element being measured is aspirated into a flame or injected into a carbon arc furnace and atomized. A light beam is directed through the flame, into a monochromator, and onto a detector that measures the amount of light absorbed by the atomized element.

Because each metallic element has its own characteristic absorption wavelength, a source lamp composed of that specific element is used to minimize spectral or radiation interferences. The amount of absorption of the characteristic wavelength is proportional to the concentration of the element in the sample.

With the EPA method, at least two types of interferences are possible: chemical and light scattering.[3,4,5,6,7] Reports on the absence of chemical interference far outnumber reports on its presence; therefore, no correction for chemical interference is given here. Non-atomic absorption as light scattering, produced by high concentrations of dissolved solids in the sample, can produce a significant interference, especially at low lead concentrations. The interference is greater at the 217.0-nm wavelength than at the 283.3-nm wavelength; in fact, Scott, D.R., et al. have reported that no interference was observed using the 283.3-nm wavelength.[3]

In this type of photometric analysis, the concentration of the sample and particularly the concentration of reagent and standard solutions, are of utmost importance to the accuracy of the determination. Samples and standard metal solutions must be carefully and accurately prepared.

5.3 Apparatus

5.3.1 Atomic Absorption Spectrophotometer - An atomic absorption spectrophotometer is required for determination of lead content in suspended particulate matter, and must be equipped with a lead hollow cathode lamp or electrodeless discharge lamp.

5.3.2 Acetylene - The grade recommended by the instrument manufacturer should be used as a fuel. Change cylinder when pressure drops below 50 - 100 psig.

5.3.3 Air - Filtered clean air (as free of particulates, oil and water as possible) is needed as an oxidant.

5.3.4 Glassware -

a. Beaker - Beakers, Borosilicate glass, including 30 ml and 150 ml are needed to digest the sample. Phillips beakers are useful for this purpose.

b. Volumetric flask - 100-ml volumetric flasks (Class A) are required for analysis.

c. Pipette - Several volumetric pipettes (Class A), including 1, 2, 4, 8, 15, 30, 50 ml should be available for the analysis.

All glassware should be thoroughly cleaned with laboratory detergent, rinsed, soaked for 4 h in 20% (w/w) HNO_3, rinsed 3 times with distilled deionized water, and dried in a dust free manner.

5.3.5 Hot plate - Hot plates, 750 watts, 120 volt, having enough plate surface area for several sample beakers, and capable of heating to 370°C (698°F) are required for the hot extraction of the sample.

5.3.6 Ultrasonication Bath Unheated - An ultrasonication bath is required for the sample extraction when an ultrasonic extraction procedure is employed, and should provide the necessary energy (>20,000 cycles per second). Commercially available baths of 450 watts or higher cleaning power have been found satisfactory.

5.3.7 Template - Templates are needed to aid in sectioning the glass fiber filter (Figure 5.1).

5.3.8 Pizza Cutter - A pizza cutter having a thin wheel (<1 mm thickness) is needed to cut the filter. (Figure 5.2).

5.3.9 Watch glass - A watch glass is needed to cover the beaker containing the sample.

5.3.10 Polyethylene Bottle - Linear polyethylene bottles are needed to store the samples for subsequent analysis.
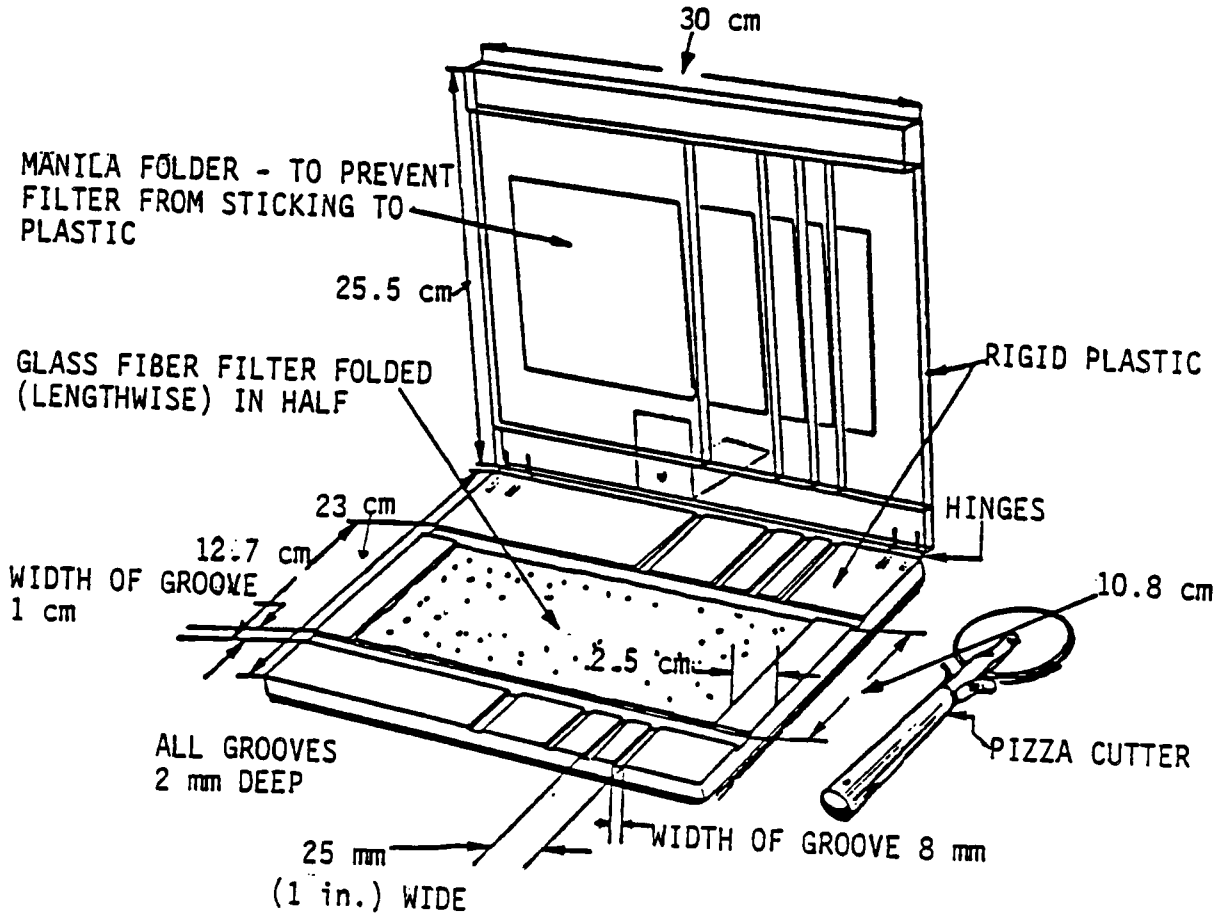
Figure 5.1. Sample preparation, filter sectioning 1.

1 mm
SLIT

STRIP FOR
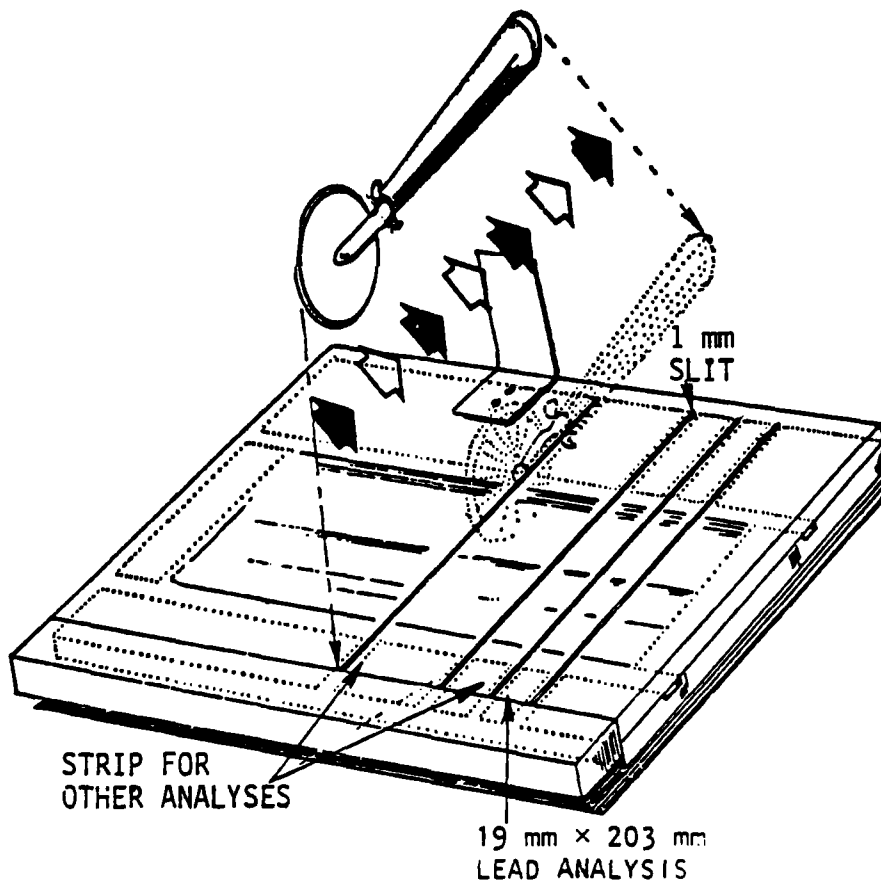OTHER ANALYSES

19 mm × 203 mm
LEAD ANALYSIS

Figure 5.2.   Sample preparation, filter sectioning 2.

5.3.11 <u>Parafilm M Sealing Film</u> - A pliable, selfsealing, moisture proof, thermoplastic sheet material, substantially colorless is recommended for use in sealing the acidified sample beakers. Commercially available Parafilm M satisfies this requirement.

5.4 <u>Reagents (Analysis)</u>

5.4.1 <u>Nitric Acid (HNO_3) Concentrated</u> - A.C.S. reagent grade HNO_3 and commercially available redistilled HNO_3 have been found to have sufficiently low lead concentration.

5.4.2 <u>Hydrochloric Acid (HCl) Concentrated</u> - A.C.S. reagent grade.

5.4.3 <u>Water</u> - The same source or batch of distilled deionized water must be used for all purposes in the analysis.

5.4.4 <u>3M HNO_3</u> - This solution is used in the hot extraction procedure. To prepare, add 192 ml of concentrated HNO_3 to distilled deionized water in a 1-ℓ volumetric flask. Shake well, cool, and dilute to volume with distilled deionized water. <u>Caution</u>: Nitric acid fumes are toxic. Prepare in a well ventilated fume hood.

5.4.5. <u>Glass Fiber Filter</u> - Low lead content of the filter is desirable. EPA typically obtains filters with a lead content of <75 µg/filter. Minimal variation in lead content from filter to filter is also important.

5.4.6 <u>0.45M HNO_3</u> - This solution is used as the matrix for calibration standards when using the hot extraction procedure. To prepare, add 29 ml of concentrated HNO_3 to distilled deionized water in a 1-ℓ volumetric flask. Shake well, cool, and dilute to volume with distilled deionized water.

5.4.7 <u>2.6M HNO_3 + (0 to 0.9M HCl)</u> - This solution is used in the ultrasonic extraction procedure and the concentration of HCl can be varied from 0 to 0.9$\underline{M}$. Directions for preparing a 2.6$\underline{M}$

HNO_3 + 0.9$\underline{M}$ HCl solution are as follows: place 167 ml of concentrated HNO_3 into a 1-ℓ volumetric flask and add 77 ml of concentrated HCl. Stir 4 to 6 h, dilute to nearly 1 ℓ with distilled deionized water, cool to room temperature, and dilute to 1 liter.

5.4.8 <u>0.40M HNO_3 + xM HCl</u> - This solution is used as the matrix for calibration standards when using the ultrasonic extraction procedure. To prepare, add 26 ml of concentrated HNO_3, plus the ml of HCl required (Equation 5-1), to a 1-ℓ volumetric flask. Dilute to nearly 1 ℓ with distilled deionized water, cool to room temperature, and dilute to 1 ℓ. The amount of HCl required can be determined from the following equation:

$$y = \frac{(77 \text{ ml}) (0.15) x}{0.9 \text{ M}} \qquad \text{Equation 5-1}$$

where

y = ml of concentrated HCl required,

x = molarity of HCl from Subsection 5.4.7, and

0.15 = dilution factor from Subsection 5.5.2.

5.4.9 <u>Lead Nitrate $Pb(NO_3)_2$</u> - A.C.S. reagent grade purity of 99.0%. Heat for 6 h at 120°C and cool in a desiccator.

5.4.10 <u>Stock Lead Solution (1000 µg Pb/ml) in HNO_3</u> - Dissolve 1.598 g of $Pb(NO_3)_2$ in 0.45$\underline{M}$ HNO_3 contained in a 1-ℓ volumetric flask and dilute to volume with 0.45$\underline{M}$ HNO_3.

5.4.11 <u>Stock Lead Solution (1000 µg Pb/ml) in (HNO_3/HCl)</u> - Prepare as in 5.4.10 except use the HNO_3/HCl solution from 5.4.8. Store standard in a linear polyethylene bottle. Commercially available certified lead standard solutions may be used. This stock solution may be stored up to 2 years. Label clearly with contents, concentration, person who prepared the standard, date prepared and expiration date. This date should be periodically checked and a fresh standard made as required.

5.5   Sample Preparation for Atomic Absorption Spectrophotometry

5.5.1   Hot Extraction Procedure -

1.   Cut a 1.9 cm × 20.3 cm (3/4 in. × 8 in.) strip from the exposed filter using a template and a pizza cutter as described in Figures 5.1 and 5.2. Other cutting procedures may be used. Care should be taken to avoid cross-contamination from one filter to another by wiping off any fibers which may adhere to template or pizza cutter between samples. Note: Lead in ambient particulate matter collected on glass fiber filters has been shown to be uniformly distributed across the filter.[3,5,8] Another study[9] has shown that when sampling near a roadway, strip position contributes significantly to the overall variability associated with lead analysis. Therefore, when sampling near a roadway, additional strips should be analyzed to minimize this variability.

2.   Fold the sample in half twice and place in a 150-ml beaker. Add 15 ml of 3$\underline{M}$ HNO$_3$ to completely cover the sample. Cover the beaker with a watch glass. It is important to keep the sample covered so that corrosion products (formed on fume hood surface which may contain lead) are not deposited in the extract.

3.   Gently boil the sample in a beaker on a hot plate under a fume hood for 30 min. Do not let the sample evaporate to dryness. Caution: Nitric acid fumes are toxic.

4.   After 30 min, remove the beaker from the hot plate and cool to near room temperature. Rinse watch glass and sides of beaker with distilled deionized water.

5.   Decant extract and rinsings into a 100-ml volumetric flask, and add distilled deionized water to 40 ml mark on beaker, cover with watch glass, and set aside for a minimum of 30 min. This is a critical step and cannot be omitted since it allows the HNO$_3$ trapped in the filter to diffuse into the rinse water.

6.   Decant the water from the filter into the volumetric flask and rinse filter and beaker twice with distilled deionized water and add rinsings to volumetric flask until total volume is 80 to 85 ml.

7.   Stopper flask and shake vigorously, and set aside for approximately 5 min or until foam has dissipated.

8.   Bring solution to volume with distilled deionized water and mix thoroughly. Allow solution to settle for one hour before proceeding with analysis. (Note: Do not filter the extracted sample to remove particulate matter because of loss of lead due to filtration. The final extract can be centrifuged at 2000 RPM for 30 min to remove any suspended solids.

9.   If sample is to be stored for subsequent analysis, transfer to a linear polyethylene bottle, being careful not to disturb the settled solids.

5.5.2   Ultrasonic Extraction Procedure -

1.   Cut a 1.9 cm × 20.3 cm (3/4 in. × 8 in.) strip, as described in Subsection 5.5.1, step 1.

2.   Fold the sample in half twice and place in a 30-ml beaker. Add 15 ml of HNO_3/HCl solution (see Subsection 5.4.7) to completely cover the sample, and cover the beaker with Parafilm. The Parafilm should be placed over the beaker such that none of the Parafilm is in contact with water in the ultrasonic bath. Otherwise, rinsing of the Parafilm in step 4 may contaminate the sample.

3.   Place the beaker in the ultrasonication bath and operate for 30 min.

4.   Rinse Parafilm and sides of beaker with distilled deionized water.

5.   Decant the extract and rinsings into a 100-ml volumetric flask. Add 20 ml distilled deionized water to cover the filter strip, cover with Parafilm, and set aside for a minimum of 30 min. This is a critical step and cannot be omitted. The sample is then processed as in Subsection 5.5.1, steps (6)

through (9). Samples prepared by hot extraction procedure are now in $0.45\underline{M}$ HNO_3 and samples prepared by ultrasonic extraction procedure are now in $0.40\underline{M}$ HNO_3 + $x\underline{M}$ HCl.

5.6 Instrument Operation and Analysis

Because of the differences between makes and models of atomic absorption spectrophotometers, it is difficult to formulate detailed instructions applicable to every instrument. Consequently, it is recommended that the user follow manufacturer's operating instructions.

1. Set the atomic absorption spectrophotometer for the standard conditions as follows: choose the correct hollow cathode lamp or electrodeless discharge lamp for lead, install, and align in the instrument; position the monochromator at 217.0 nm or 283.3 nm; select the proper monochromator slit width; set the light source current according to the manufacturer's recommendation; light the flame and regulate the flow of fuel and oxidant; adjust the burner for maximum absorption and stability; and balance the photometer.

2. If using a chart recorder set the chart speed at 8 cm to 15 cm per minute and turn on the power, servo, and chart drive switches. Adjust the chart pen to the 5% division line. Also adjust instrument span using highest calibration standard. While aspirating the standard sample, span instrument to desired response.

3. The sample can be analyzed directly from the volumetric flask, or an appropriate amount of sample decanted into a sample analysis tube. In either case, care should be taken not to disturb the settled solids. At least the minimum sample volume required by the instrument should be available for each aspiration.

4. Aspirate samples, standards, and blank into the flame and record the absorbance. If using a recorder wait for response to stabilize before recording absorbance.

5.   Determine the average absorbance value for each known concentration, and correct all absorbance values by subtracting the blank absorbance value. Determine the lead concentration in µg Pb/ml from the calibration curve as presented in the following subsection. Record these values on the Data Record Form (Figure 6.1 of Section 2.8.6).

Note:

a.   Samples that exceed the calibration range should be diluted with acid of the same concentration and matrix as the calibration standards and reanalyzed.

b.   Check for drift of the zero point resulting from possible nebulizer clogging, especially when dealing with samples of low absorbance.

5.7   Preparation of Calibration Curve

5.7.1   Working Standard Solution (20 µg Pb/ml) - Prepare by diluting 2 ml of stock lead solution (Subsection 5.4.10 if the hot extraction was used or Subsection 5.4.11 if the ultrasonic extraction procedure was used) to 100 ml with acid of the same concentration and matrix as used in the stock lead solution. This standard should be prepared daily.

5.7.2   A Series of Calibration Standards - Prepare daily by diluting the working standard solution (Subsection 5.7.1) as indicated below with the same acid matrix as used in the working solution. Other lead concentrations may be used provided they are in the linear range of the instrument.

Volume of 20 µg/ml working standard, ml	Final volume, ml	Concentration µg Pb/ml
0	100	0.0
1.0	200	0.1
2.0	200	0.2
2.0	100	0.4
4.0	100	0.8
8.0	100	1.6
15.0	100	3.0
30.0	100	6.0
50.0	100	10.0
100.0	100	20.0

5.7.3 <u>Calibration Curve</u> - Since the working range of analysis, will vary depending on which wavelength is used and the type of instrument, no one set of instructions for preparation of a calibration curve can be given.

Select at least three standards (plus the reagent blank) to cover the linear range indicated by the instrument's manufacturer. Aspirate these standards and the blank and measure the absorbance. Repeat until good agreement is obtained between replicates. Plot absorbance (y-axis) versus concentration in µg Pb/ml (x-axis) as shown in Figure 5.3. Draw a straight line through the linear portion of the curve, and do not force the calibration curve through zero. <u>Note</u>: To determine stability of the calibration curve, analyze a control standard before the first sample, after every subsequent 10th sample, and after the last sample. Vary the control standard concentration by alternating, in run sequence, a value less than 1 µg Pb/ml, and a value between 1 and 10 µg Pb/ml. If either standard deviates by more than 5% from the value predicted by the calibration curve, take corrective action and repeat the previous 10 analyses.
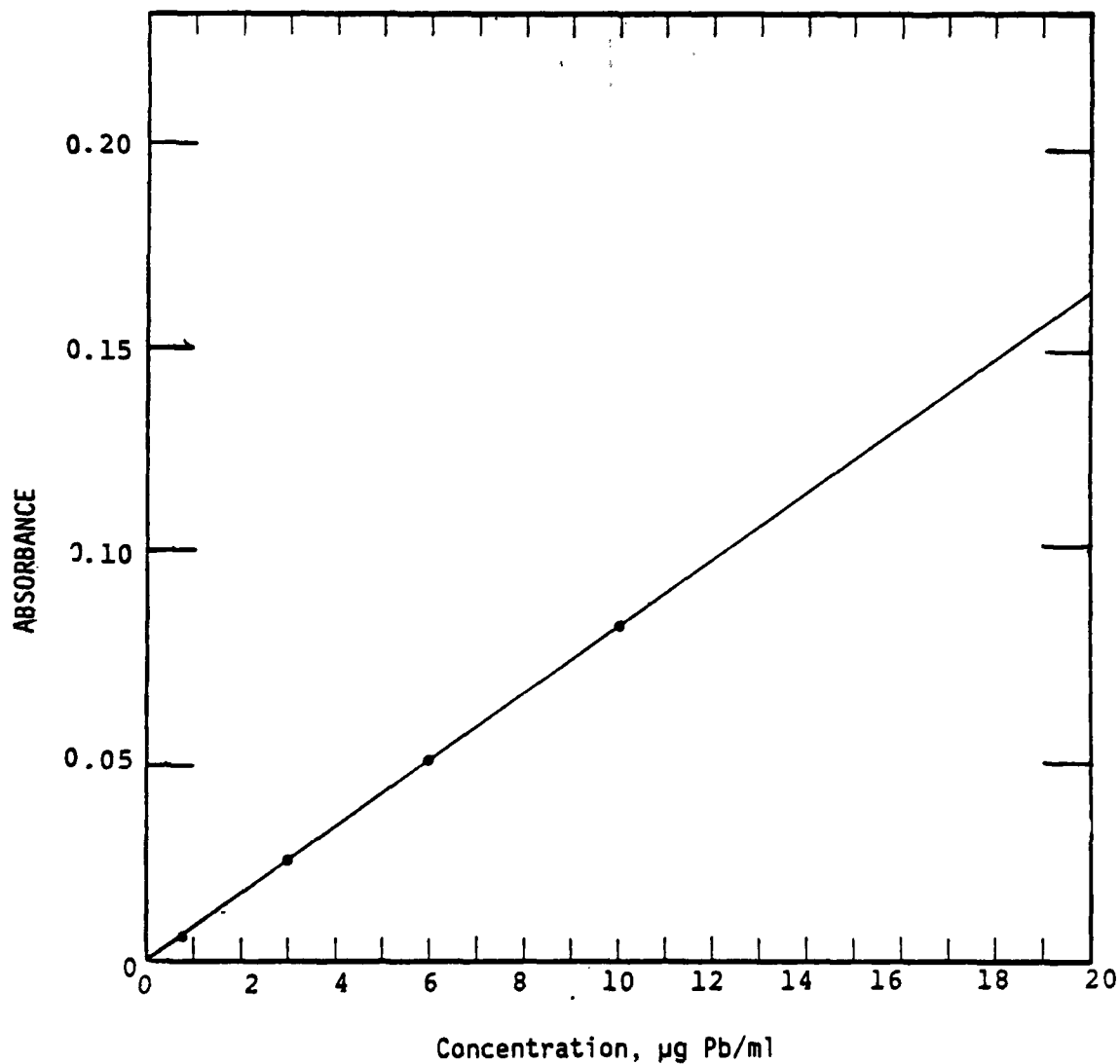
Figure 5.3. Example of a calibration curve for absorbance versus concentration of Pb standard.

TABLE 5.1.   ACTIVITY MATRIX FOR ANALYSIS OF SAMPLES

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Verify documentation and inspect sample	Complete documentation; absence of evidence of malfunction or sample loss; ten or fewer insects visible in sample	Visual check	Void sample
Atomic absorption spectrophotometer	Equipped with lead hollow cathode lamp or electrodeless discharge lamp	Upon receipt check for specifications or certification	Service by manufacturer
Reagents	All reagents must be A.C.S. reagent grade	Prepare fresh as introduced in Subsec 5.4	Use new reagents
Glassware	Borosilicate glass and Class A	Upon receipt check for stock number, cracks, breaks, and manufacturer flaws	Replace or return to supplier
Filter strip	Size = 1.9 cm × 20.3 cm (3/4 in. × 8 in.)	Check its size	Prepare new strip
Hot extraction	Do not evaporate to dryness and cover so that corrosion products are not deposited in the extract	Frequently and visually check the level of evaporation	Void sample
Ultrasonic extraction	Provide >20,000 cycles per second	Upon receipt check the label and performance by comparing to the hot extraction procedure	Return to supplier or use a hot extraction procedure
Sample acid concentration	$0.45\underline{M}$ HNO_3; or $0.40\underline{M}$ HNO_3 + $x\underline{M}$ HCl	Prepare fresh	Void sample
Calibration curve	Reproducibility is ±5%	Recalibrate and repeat the analysis	Check instrument or prepare a new calibration curve

6.0 CALCULATIONS AND DATA REPORTING

A matrix summarizing the quality control activities for the calculations and the data-reporting requirements is presented in Table 6.1.

6.1 Sample Air Volume

At standard temperature and pressure (STP) for samplers equipped with rotameters:

$$V = \frac{(Q_i + Q_f)}{2} t \qquad \text{Equation 6-1}$$

where

V = air volume sampled, m^3,

Q_i = initial air flow rate, m^3/min at STP,

Q_f = final air flow rate, m^3/min at STP, and

t = sampling period (elapsed time), min.

For samplers equipped with flow recorders:

$$V = Qt \qquad \text{Equation 6-2}$$

where Q = average sampling rate, m^3/min at STP.

Estimate the Q from the recorder chart. If the flow rate varies less than 0.11 m^3/min during the sampling period, read the flow rate from the chart at 2-h intervals and take the average value for Q.

Calculation for sample air volume is identical to the hi-vol method.

6.2 Lead Concentration

6.2.1 Estimation of Lead Concentration of the Blank Filter, µg -

1. For testing of the large batches of filters (>500 filters) select at random 20 to 30 filters from a given batch. For small batches (<500 filters) a lesser number of filters may be taken. Cut one 1.9 cm × 20.3 cm (3/4 in. × 8 in.) strip from

each filter, anywhere in the filter. Analyze all strips, sepa-
rately, according to the directions in Subsections 5.5 and 5.6
of Section 2.8.5.

    2.   Calculate total lead in each filter as

$$F_b = \mu g \ Pb/ml \times \frac{100 \ ml}{strip} \times \frac{12 \ strips}{filter}$$

where

        F_b = Amount of lead per 465 square cm (72 square
           in.) of blank filter, μg,

   $\mu g \ Pb/ml$ = Lead concentration determined from Subsection
           5.6 of Section 2.8.5,

  100 ml/strip = Total sample volume,

12 strips/filter =

$$\frac{Useable \ filter \ area, \ 20 \ cm \times 23 \ cm \ (8 \ in. \times 9 \ in.)}{Exposed \ area \ of \ one \ strip, \ 1.9 \ cm \times 20 \ cm \ (3/4 \ in. \times 8 \ in.)} .$$

    3.   Calculate the mean, $\bar{F}_b$, and the relative standard de-
viation (100 × standard deviation/mean).

$$\bar{F}_b = \frac{\sum\limits_{1}^{n} F_{b_i}}{n} \qquad\qquad\qquad \text{Equation 6-3}$$

where

        $\bar{F}_b$ = average amount of lead per 72 square inches of
           filter, μg

        F_{b_i} = amount of lead per 72 square inches for each
           filter, μg

        n = number of blank filters analyzed.

The standard deviation (SD) of the analyses for the blank fil-
ters is given by Equation 6-4,

$$SD = \left[\frac{\sum\limits_{1}^{n} (F_{b_i} - \bar{F}_b)^2}{n-1} \right]^{1/2} . \qquad\qquad \text{Equation 6-4}$$

The relative standard deviation (RSD) is the ratio

$$RSD = \frac{100 \; SD}{\bar{F}_b}.$$

If the relative standard deviation is high enough so that in the analyst's opinion subtraction of $\bar{F}_b$ may result in a significant error in the $\mu g \; Pb/m^3$, the batch should be rejected. For acceptable batches, use the value of $\bar{F}_b$ to correct all lead analyses (Subsection 6.2.2) collected using that batch of filters. If $\bar{F}_b$ is below the lower detectable limit (LDL), no correction is necessary.

6.2.2 <u>Calculation of Lead Concentration of the Exposed Filter</u> - Lead concentration in the air sample can be calculated from data tabulated on data record form (Figure 6.1) as follows:

$$C = \frac{(\mu g \; Pb/ml \times 100 \; ml/strip \times 12 \; strips/filter) - \bar{F}_b}{V}$$

where

$$C = \text{Concentration, } \mu g \; Pb/m^3,$$

$\mu g \; Pb/ml$ = Lead concentration determined from Section 2.8.5, Subsection 5.6,

100 ml/strip = Total sample volume,

12 strips/filter =

$$\frac{\text{Useable filter area, 20 cm} \times \text{23 cm (8 in.} \times \text{9 in.)}}{\text{Exposed area of one strip, 1.9 cm} \times \text{20 cm (3/4 in.} \times \text{8 in.)}} \; ,$$

$\bar{F}_b$ = Average lead concentration of blank filters, μg,

V = Air volume from Subsection 6.1.

6.2.3 <u>Sample Calculation of Lead Concentration in Air Sample</u> - Data are tabulated on a data record form as shown in Figure 6.1. The standard data are recorded and a standard curve is drawn (Figure 6.2). The line of best fit is drawn through the points. Average all standards analyzed throughout the run but do not include the standards used as checks of the calibration stability. These are check samples to be compared to the calibration curve.

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Project _City of Cincinnati_   Date _January 2, 1981_

Sample location _AE_   Analyst _D. Albrinck_

				Sample Data	
Sample number	Air volume at STP, m³	Avg blank $\bar{F}_b$, µg	Absorbance	Concentration from cal curve, µg Pb/ml	Total Pb, µg Pb/m³
11AE	2291	<75	0.042	5.7	3.0

Standard Data

Vol. of 20 µg/ml working standard, ml	Concentration, µg Pb/ml	Absorbance
0	0.0	0.000
8.0	1.6	0.013
15.0	3.0	0.023
30.0	6.0	0.049
50.0	10.0	0.084
100.0	20.0	0.156

Checked by _Douglas J. Morell_

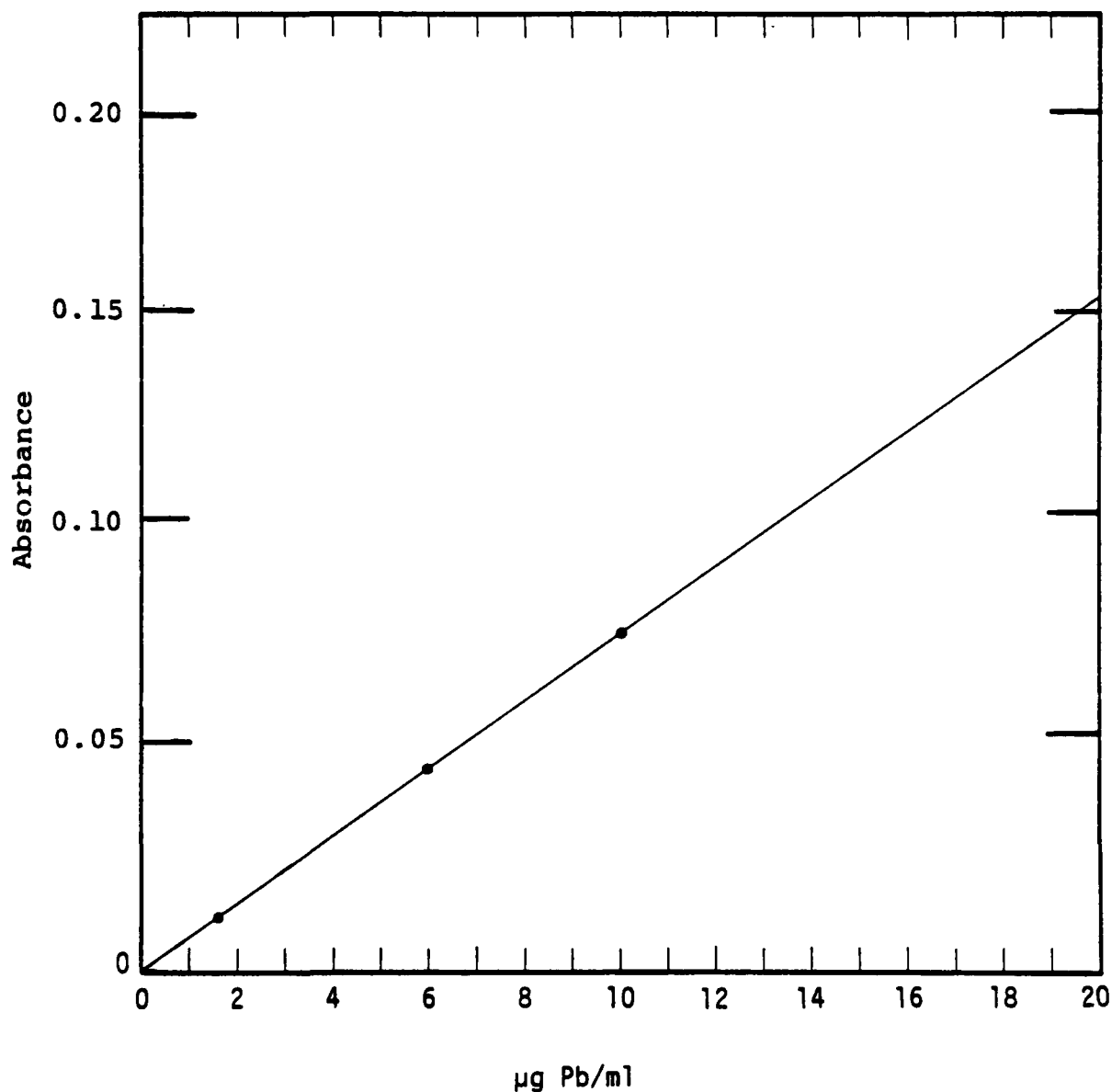Figure 6.1. Example of a data record form.

Figure 6.2.   Calibration curve.

To calculate total Pb of a sample use equation from Subsection 6.2.2.

$$C = \frac{[(5.7 \text{ } \mu g/ml)(100 \text{ } ml/strip)(12 \text{ } strip/filter)] - (0)}{(2291 \text{ } m^3)}$$

$$C = 3.0 \text{ } \mu g \text{ } Pb/m^3.$$

TABLE 6.1. ACTIVITY MATRIX FOR CALCULATION AND DATA REPORTING

Activity	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Calculations (1) sample volume (2) $\overline{F}_b$, blank (3) SD and RSD of F_b values (4) sample concentration	All needed data available; relative standard deviation of $\overline{F}_b$ is not high	Visual check for each sample; repeat all calculations	Void sample; indicate errors and make corrections
Analysis data form	All data and calculations are given	Visual check	Complete missing data values
Documentation and sample verification	Documentation complete for calculation of concentration; all sample and data identification numbers match; absence of evidence of malfunction or sample loss	Visual check for each sample	Void sample
Documentation of report data	All needed data available	Visual check for each sample	Void sample

7.1 Atomic Absorption Spectrophotometer

As previously indicated, major maintenance and calibration should be done by service engineers or qualified operators. The following general maintenance procedures should be carried out only after consulting the manufacturer's manual.

7.1.1 Light Source - When problems are concerned with a light source, check the hollow cathode lamp or electrodeless discharge lamp mounting bracket, lamp connection, and make sure the instrument is plugged in, turned on, and warmed up. If line voltages are low, operate the power supply from a variac which is set to give maximum voltage. Lamp current meter fluctuation can be reduced by using a constant voltage sine wave transformer.

7.1.2 No Absorbance Response - Make sure that the lamp is lighted, properly aligned, and that the wavelength, slit, and range controls are properly adjusted. If the meter cannot be zeroed, (1) adjust the level of the burner head to avoid intercepting the light beam, and (2) clean the lamp and window, or photometer cover windows, with a dilute solution of a mild

detergent and rinse several times with distilled water. Dirty windows or lenses are a major problem when operating the instrument below 2300 A° (230 nm).

7.1.3 <u>Readout Noisy, Flame On</u> - Check the lamp current setting, fuel and oxidizer flow rates, the <u>leviner</u> to make sure it is draining properly, the nebulizer for corrosion around the tip, the adjustment of the nebulizer capillary, the burner head (it may need cleaning with razor blade), the acetylene cylinder pressure, the air pressure, and the air line filter.

7.1.4 <u>Poor Sensitivity (Within 50% of That Suggested in the</u> <u>Analytical Method Book)</u> - Check the sensitivity obtainable for several other elements to ascertain that the low sensitivity is not due to the lamp used. Check the slit width, wavelength, range setting, the burner alignment, the adjustment of the nebulizer capillary, the fuel/oxidant flow rate ratio to ascertain that it is optimized for the element to be analyzed. Make sure that the lamp current is not above the recommended value, check the lamp alignment, and the concentration of the standard solution used.

All other maintenance problems such as cleaning of mirrors or gratings should be discussed with the manufacturer or service representative.

8.0 AUDITING PROCEDURE

An audit is an independent assessment of the quality of data. Independence is achieved by having the audit made by an operator other than the one conducting the routine field measurements and by using audit standards and equipment different from those routinely used. Routine quality assurance checks conducted by the operator are necessary for obtaining and reporting good quality data, but they are not to be considered as part of the auditing procedure.

Based on the results of the Reference Method Test[10] for lead analysis and Hi-Vol Sampling Method[2], three performance audits and a system audit are recommended and are described in detail in the subsequent sections.

The basic purpose of an auditing program is to ensure the integrity of the data and to assess the data in terms of accuracy. Techniques for estimating the accuracy of the data are given in Section 2.0.8 of Volume II of this Handbook.

8.1 Performance Audits

Performance audits are independent checks made by the supervisor or auditor to evaluate the quality of data produced by the total measurement system (sample location, sample analysis and data processing). Performance audits are normally a quantitative appraisal of quality.

Three performance audits of individual variables are recommended:

1. Audit of flow rate calibration
2. Audit of lead analysis
3. Audit of data processing.

8.1.1 Audit of Flow Rate Calibration - The frequency of the audit of the flow rate depends on the use of the data (e.g., for PSD air monitoring or for SLAMS). It is recommended that the flow rate of each hi-vol sampler be audited each quarter.

1. Conduct the flow rate audit using a reference flow (ReF) device,[a] or a similar device.

2. Audit the flow rate at one flow rate. The ReF device used for auditing must be different from the one used to calibrate the flow of the hi-vol sampler being audited.

3. Operate the hi-vol sampler at its normal flow rate with the audit device in place.

4. Great care must be used in auditing the hi-vol samplers having flow regulators because the introduction of resistance plates in the audit device can cause abnormal flow patterns at the point of flow sensing. For this reason, the orifice of the flow audit device should be used with a normal glass fiber filter in place and without resistance plates in auditing flow regulated hi-vol samplers, or other steps should be taken to assure that flow patterns are not disturbed at the point of flow sensing.

5. Use the known audit flow measurement and the flow measured by the sampler's normal flow indicator to calculate percent difference (Equation 8-1), a measure of inaccuracy. Both flows must be referenced to same temperature and pressure. Let X_i represent the known flow rate, Y_i the measured flow rate, and d_i the percent difference for the ith audit:

$$d_i = \frac{Y_i - X_i}{X_i} \, 100. \qquad \qquad \text{Equation 8-1}$$

Thus if Y_i = 52 ft^3/min and X_i = 50 ft^3/min, then

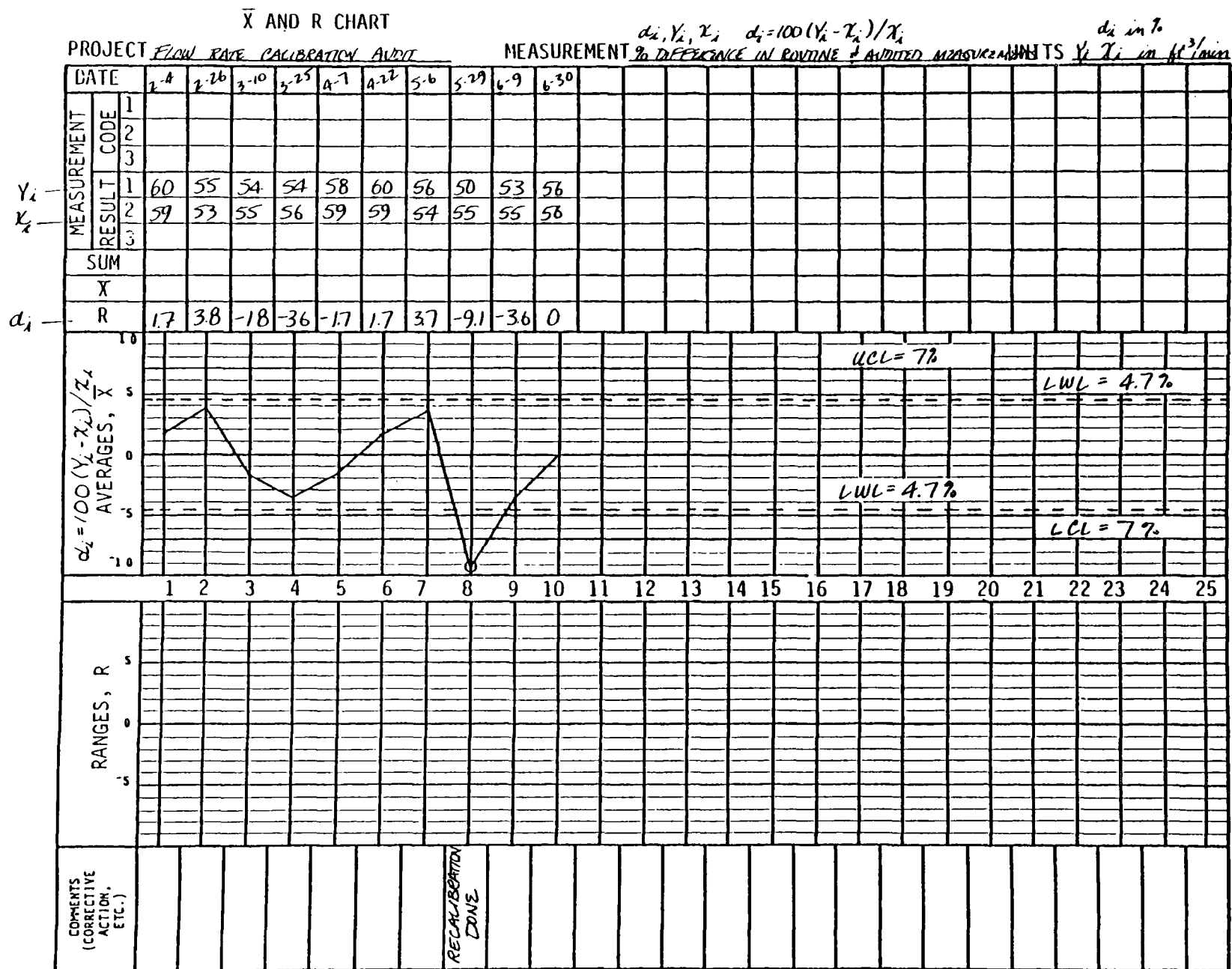$$d_i = \frac{52 - 50}{50} \, 100 = 4\%.$$

If d_i is greater than ±7% for any one check, recalibrate before resuming the sampling.

---

[a]USEPA uses ReF device with five orifice plates that mount onto the faceplate of the hi-vol adaptor; this device may be purchased from Dexco, Co., Inc., 630 Chapel Hill Blvd., Burlington, N. C. 27215.

6.   Report the Y_i, the X_i, and the d_i on an $\bar{X}$-and-R chart (Figure 8.1) under "Measurement Result, Items 1 and 2." Record the d_i in the cells preceded by the "Range R." The d_i can be positive or negative, but the range is always positive; so retain the sign of the difference since it may indicate trends and/or consistent biases. The steps in the construction of a quality control chart and the interpretation of the results are in Appendix H, Volume I of this Handbook.[11]

7.   Repeat the above for each flow rate calibration audit; plot all points on the chart; and connect the points by drawing a straight line. Tentative limits are ±4.7% (warning lines) and ±7% (out-of-control lines). Out-of-control points indicate possible problems in calibration errors or instrument damage. Recalibrate the sampler prior to further sampling when out of control. After 15 to 20 points are plotted, new control and warning limits may be derived, as described in Appendix H of Volume I of this Handbook.[11] Do not increase the control and warning limits, however, more stringent limits may be established.

8.1.2 <u>Audit of Lead Analysis</u> - Each calendar quarter, audit the lead analysis using glass fiber filters containing a known quantity of lead. Audit samples are prepared by depositing a lead solution Pb $(NO_3)_2$, on 1.9 cm × 20 cm (3/4 in. by 8 in.) unexposed glass fiber filter strips, and allowing to dry thoroughly. It is required that the audit samples be prepared using reagents different from those used to generate the lead calibration curve in Section 2.8.5. If the routine network operators are used to perform the audit, these operators must not know the audit values prior to the audit. This means another individual must administer the audit program. Prepare blind audit samples in the following concentration ranges:

X̄ AND R CHART

PROJECT _FLOW RATE CALIBRATION AUDIT_   MEASUREMENT _% DIFFERENCE IN ROUTINE & AUDITED MEASUREMENTS_ d_i, Y_i, X_i $d_i = 100(Y_i - X_i)/X_i$ UNITS _Y_i X_i in ft³/min_ d_i in %

DATE			2-4	2-26	3-10	3-25	4-7	4-22	5-6	5-29	6-9	6-30															
MEASUREMENT	CODE	1																									
		2																									
		3																									
	RESULT Y_i X_i	1	60	55	54	54	58	60	56	50	53	56															
		2	59	53	55	56	59	59	54	55	55	56															
		3																									
	SUM																										
	X̄																										
d_i	R		1.7	3.8	-1.8	-3.6	-1.7	1.7	3.7	-9.1	-3.6	0															

UCL = 7%
LWL = 4.7%
LWL = 4.7%
LCL = 7%

$d_i = 100(Y_i - X_i)/X_i$ AVERAGES, X̄

RANGES, R

COMMENTS (CORRECTIVE ACTION, ETC.)   RECALIBRATION DONE

Figure 8.1.   Quality control chart for flow rate calibration audit.

Range	Conc. µg Pb/strip	Conc. µg Pb/m³*
1	100 to 300	0.5 to 1.5
2	600 to 1000	3.0 to 5.0

*Calculation of lead concentration in µg/m³ is based on sampling at 1.7 m³/min for 24 h on an 20 cm × 25 cm (8 in. × 10 in.) glass fiber filter.

Analyze at least one audit sample in each of the two ranges each day that samples are analyzed. If samples are analyzed only once per quarter, analyze at least two audit samples in each of the two ranges. The percentage difference d between the audit concentration (µg Pb/strip) and the analyst's measured concentration (µg Pb/strip) is used to calculate analysis inaccuracy, (Equation 8-2),

$$d = \frac{C_{Pb(M)} - C_{Pb(A)}}{C_{Pb(A)}} \times 100 \qquad \text{Equation 8-2}$$

where

d = Percentage difference,

$C_{Pb(M)}$ = Concentration measured by the lab analyst, µg Pb/ml, and

$C_{Pb(A)}$ = Audited or known concentration of audit sample, µg Pb/ml.

Tabulate the percentage differences on an $\bar{X}$ and R chart (optional). The upper and lower control lines and the upper and lower warning lines should also be plotted (Figure 8.2) and used as guidelines to determine when results are questionable and corrective action needs to be taken. Record on the chart the nature of the corrective action. Details for construction of a quality control chart are given in Appendix H of Volume I of this Handbook.[11]

The recommended control limits for the two audit sample ranges (0.5 to 1.5 and 3 to 5 µg Pb/m³) are the 90th percentile values for d based on the results of seven audits (8/77, 1/78, 6/78, 1/79, 7/79, 1/80, and 7/80) performed by the Environmental

X and R CHART

PROJECT: A.A. analytical audit

MEASUREMENT PERFORMED: $d = [C_{Pb(M)} - C_{Pb(A)}] 100 / C_{Pb(A)}$

MEASUREMENT UNITS: Pb μg/ml, d in %

1976 DATE		1/21	1/27	2/3	2/9	3/15	3/21	3/27	3/5	3/11	3/17	3/23	3/27	4/4
RESULT CODE	1													
	2													
	3													
$C_{Pb(A)}$	1	6.0	6.0	6.0	6.0	6.0	6.0	6.0	60	60	60	6.0	6.0	6.0
$C_{Pb(M)}$	2	5.8	5.9	6.1	60	6.2	5.7	60	5.9	6.3	6.1	6.8	60	5.8
	3													
SUM														
AVERAGE, X̄														
d RANGE, R		3.33	1.67	1.67	0	3.33	5.00	0	1.67	5.00	1.67	3.33	0	3.33

AVERAGES, X̄ $d = \left[\frac{C_{Pb(M)} - C_{Pb(A)}}{C_{Pb(A)}}\right](100)$

UCL = 5.22
UWL = 3.48
LWL = -3.48
LCL = -5.22

RANGES, R

Comments (Correct Action, etc.)

Figure 8.2.   Quality control chart for A.A. analytical audit.

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Monitoring Systems Laboratory, USEPA, Research Triangle Park, North Carolina.[12,13,14,15] By definition, 90% of the laboratory participants in the audit obtained values of d less than the values tabulated below. The control limits are expected to be exceeded by 10% of the laboratories to be audited, based on these seven audits over four years. The 90th percentile values and the known audit concentrations are given below for each audit concentration range.

0.5 to 1.5 µg Pb/m³

Audit date	Known audit concentration, µg Pb/m³	90th percentile for d, %
8/77	1.8	45
1/78	0.6	35
6/78	0.4	31
6/78	1.5	15.3
1/79	1.5	15.1
7/79	1.2	16
1/80	0.9	16.1
7/80	0.6	11.7

3 to 5 µg Pb/m³

Audit date	Known audit concentration, µg Pb/m³	90th percentile for d, %
8/77	4.0	23.5
6/78	5.0	12.5
1/79	3.5	14.8
7/79	3.5	13.9
1/80	4.5	20.0
7/80	4.2	9.8

Based on the results of these seven audits, the recommended 90th percentile control limits for audit samples are ±16% for both the 0.5 to 1.5 µg Pb/m³ and the 3.0 to 5.0 µg Pb/m³ concentration ranges. The control limits of ±16% are also recommended

for the lower Pb concentrations range (0.5 to 1.5 µg Pb/m^3) on the assumption that the first three audits are not representative of current limits (last four audits).

The method user should take part in the EPA semi-annual audit program for lead analysis. For more information on the EPA lead audit program, see Section 2.0.10 of this Handbook.

8.1.3 <u>Audit of Data Processing</u> - A data processing audit allows for correction of errors after the original calculations have been performed. The audit rate of seven measurements out of every 100 is recommended. The audit is made starting with the raw data on the data form. When the original and the audit calculations do not agree, all calculations for the corresponding audit period should be recalculated. The nature of the error(s) should be clearly explained to the appropriate personnel in order to minimize their reoccurrence. Audit values are recorded in the data log and reported to the supervisor for review. These results can be used to check computer programs and manual methods of data processing.

8.2 <u>System Audit</u>

A system audit is an on-site inspection and review of the quality assurance system used for the total measurement system (sample collection, sample analysis, data processing, etc.). Whereas performance audits are a quantitative assessment, system audits are normally a qualitative appraisal.

A system audit should be conducted for a monitoring system. The auditor should have sufficient experience with the method and an extensive background with the characterization technique that he is auditing. Figure 8.3 can be used as a preliminary form for use in a system audit. These questions should be checked for the applicability to the particular local, State, or Federal agency. One should also refer to Section 2.0.11 of this volume of the Handbook for further details on a system audit.

1. What types of hi-vol samplers are utilized in the network?
    _____General motor Works_____Model H____

2. How often are the samplers run? ((a)) daily, (b) once every six days, (c) once every 12 days, (d) other_____

3. What type and quality of filter and number of filters is being utilized?
    ___glass fiber, spectroquality grade._____

4. Are there any pre-exposure checks for pin holes or imperfections run on the filters? _____yes_____

5. What is the collection efficiency for your filters? ___99 %____

6. What is the calibration procedure for the hi-vol sampler?
    __Calibration Procedure outlined in Section 2.2, Subsection 2

7. Which statement most closely estimates the frequency of flow rate calibration? (a) once when purchased, (b) once when purchased, then after every sampler modification, or ((c)) once when purchased, then at regular intervals thereafter _____

8. Are flow rates measured before and after sampling period? Yes ✓ No __

9. If the answer to number 8 is yes using the equation below, what is the estimated average percent of change in the flow rates?

    $$\frac{100 \ (Q_i - Q_f)}{Q_i} = \text{percent change}$$

    ((a)) less than 10%, (b) 10-20%, or (c) greater than 20% _____

10. Is there a log book at each sampler to record flows and times?
    Yes ___✓___ No _____

11. Is the atomic absorption spectrophotometer properly calibrated? __yes__
    If so, when? __calibration curve run with every sample group

12. Are all components of the atomic absorption spectrophotometer correctly aligned? Yes ___✓___ No _____

13. Qualifications of atomic absorption spectrophotometer operator? _1 year experience w/ AA analysis, certified technicians

14. Extraction procedure. (1) Hot extraction ___✓___
    (2) Ultrasonic extraction _____

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15. Calibration curve check? _____ _OK_____

16. Calculation procedure check? _____ _OK_____

17. Are reagents, calibration standards, samples, etc., labeled clearly with test numbers, dates, and all pertinent data? ___ _yes_____

   Comments: _____

   _____

Figure 8.3. Checklist for use by auditor.

The system audit should be performed at the beginning of the monitoring program and annually thereafter unless problems occur to require more frequent system audits.

8.3 Activity Matrix

Table 8.1 summarizes the quality assurance activities for auditing procedures.

TABLE 8.1. ACTIVITY MATRIX FOR AUDITING PROCEDURE

Audit	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Flow rate audit	$d_i = \dfrac{Y_i - X_i}{X_i} \, 100$ Y_i = routinely measured flow rate, and X_i = audited flow rate	Audit each sampler quarterly; same method as for calibration procedure	Corrective action before resuming sampling; action noted on X- and- R chart
Audit of analysis process using audit samples	d should be within ±16% for both the 0.5 to 1.5 µg Pb/m³ and the 3 to 5 µg Pb/m³ concentration ranges (Subsec 8.1.2)	Analyze an audit sample in each of the two concentration ranges at least once each analysis day and at least twice per calendar quarter that samples are analyzed (Subsec 8.1.2)	Calibration curve checked, if necessary; a new reference sample checked, and if acceptable, analysis resumed; data accuracy calculated per Sec 2.0.8
Data processing audit	The reported value should agree with the audited value within round-off error	1 in 14 samples or 1/mo, whichever is greater; independent calculations from raw data to final recorded data	Calculations for all samples collected since previous audit checked and corrected
System audit	Method described in this section of Handbook	At the beginning of a new monitoring system, and periodically as appropriate; observation of procedures and use of a checklist	Improved methods and/ or training programs initiated

12. Bromberg, S. M., R. L. Lampe, and B. I. Bennett. Summary of Audit Performance: Measurement of SO_2, NO_2, CO, Sulfate, Nitrate, Lead and Hi-Vol Flow Rate - 1978, EPA-600/4-80-017. U.S. Environmental Protection Agency, Research Triangle Park, N.C., June 1980.

13. Bromberg, S. M., R. L. Lampe, and B. I. Bennett. Summary of Audit Performance: Measurement of SO_2, NO_2, CO, Sulfate, Nitrate, Lead, Hi-Vol Flow Rate -1977, EPA-600/4-79-014. U.S. Environmental Protection Agency, Research Triangle Park, N.C., February 1979.

14. U.S. Environmental Protection Agency. National Performance Audit Program. 1979 Proficiency Surveys for Sulfur Dioxide, Nitrogen Dioxide, Carbon Monoxide, Sulfate, Nitrate, Lead and High Volume Flow. EPA-600/ 4-81-025, April 1981.

15. Bennett, B. I., R. L. Lampe, L. F. Porter, A. P. Hines, and J. C. Puzak. National Performance Audit Program. 1980 Proficiency Surveys for Sulfur Dioxide, Nitrogen Dioxide, Carbon Monoxide, Sulfate, Nitrate, Lead and High Volume Flow. U.S. Environmental Protection Agency, Research Triangle Park, N. C., September 1981.

16. Catalog of NBS Standard Reference Materials. NBS Special Publication 260, 1982 - 83 Edition. U.S. Department of Commerce, NBS, Washington, D. C. November 1981.

Review Exercise

Now that you've completed the assignment for Section 7, please answer the following questions. These will help you determine whether you are mastering the material.

1. The lead reference method requires that particulate lead be collected by a(n) _____(?)_____ and analyzed by a(n) _____(?)_____.
   a. impinger, ultraviolet photometer
   b. dust fall bucket, atomic absorption spectrophotometer
   c. high volume sampler, atomic absorption spectrophotometer
   d. high volume sampler, flame ionization detector

2. Before using an atomic absorption spectrophotometer for lead analysis, its _____(?)_____ should be checked.
   a. sensitivity
   b. reproducibility
   c. both a and b, above

3. An atomic absorption spectrophotometer that is used to analyze lead samples should have a reproducibility change of _____(?)_____ percent or less.
   a. 1
   b. 2
   c. 5
   d. 10

4. True or False? In the lead reference method, lead atoms absorb light over a broad band of wavelengths.

5. Which of the following is a(are) possible type(s) of interference(s) in the atomic absorption analysis of lead?
   a. chemical
   b. light scattering
   c. both a and b, above

6. Acetylene cylinders used in the atomic absorption analysis of lead should be replaced when their pressures drop below _____(?)_____ psig.
   a. 25 to 50
   b. 50 to 100
   c. 100 to 150
   d. 150 to 200

7. Air used in the atomic absorption analysis of lead should be as free of
   ___(?)___ as possible.
   a. particulate matter
   b. oil
   c. water
   d. a and b, above
   e. a, b, and c, above

8. Volumetric glassware used in the analysis of lead must be Class ___(?)___.
   a. A
   b. B
   c. C

9. Which of the following should be used in cleaning glassware for lead analysis?
   a. laboratory detergent
   b. 20% (w/w) HNO_3
   c. distilled deionized water
   d. both a and c, above
   e. a, b, and c, above

10. If possible, reagents used in lead analysis should be at least ___(?)___.
    a. ACS reagent grade
    b. practical grade
    c. technical grade

11. Which of the following is a(are) desirable characteristic(s) of glass-fiber filters
    used to collect particulate lead?
    a. minimal variation of lead content from filter to filter
    b. high lead content
    c. both a and b, above

12. True or False? The same source or batch of distilled deionized water must be
    used for all purposes in lead analysis.

13. Stock lead solutions may be stored up to ___(?)___ year(s).
    a. 1
    b. 2
    c. 3
    d. 5

14. True or False? One strip of a particulate lead filter collected near a roadway
    should be analyzed to determine the ambient air lead concentration.

15. Which of the following is an(are) EPA-accepted method(s) of removing lead
    from particulate-matter filters?
    a. hot extraction procedure
    b. ultrasonic extraction procedure
    c. liquid chromatography extraction procedure
    d. both a and b, above
    e. both b and c, above

16. Which of the following should be performed during lead extraction?
    a. keep the lead sample covered during extraction
    b. allow sample filter to soak in distilled deionized water for 30 minutes
    c. filter the extracted sample to remove particulate matter
    d. a and b, above
    e. a, b, and c, above

17. True or False? In the hot extraction procedure, the lead sample should be evaporated to dryness.

18. True or False? Settled solids in a lead sample should not be disturbed when the sample is aspirated into the flame of an atomic absorption spectrophotometer.

19. True or False? Lead samples having concentrations that exceed the calibrated range of an atomic absorption spectrophotometer should be diluted with distilled deionized water.

20. Lead standard working solutions should be prepared ____(?)____.
    a. daily
    b. weekly
    c. biweekly
    d. monthly

21. At least ____(?)____ standard(s) plus a reagent blank should be used to prepare a lead calibration curve for an atomic absorption spectrophotometer.
    a. 1
    b. 2
    c. 3
    d. 5

22. True or False? Control standards should be periodically analyzed, using an atomic absorption spectrophotometer, during the analyses of lead samples.

23. If a control standard analysis deviates more than ____(?)____ percent from the value predicted by the calibration curve of an atomic absorption spectrophotometer, corrective action should be taken.
    a. 0.5
    b. 1
    c. 5
    d. 10

24. Under the conditions described below, the lead concentration obtained using the lead reference method is ____(?)____ μg/std m³.
    a. 0.826
    b. 0.044
    c. 1.00
    d. 1.04

      Given: Total volume of sample solution: 100 mL/strip
                    Lead concentration of sample solution: 2.0 μg/mL
                    Average lead concentration of blank filters: 100 μg
                    Volume of air sampled: 2300 std m³

25. True or False? Current fluctuations of lead atomic absorption lamps can be reduced by using constant voltage sine wave transformers.

26. Which of the following is a(are) possible cause(s) for being unable to "zero" an atomic absorption spectrophotometer?
    a. burner head intercepting the light beam
    b. dirty spectrophotometer windows
    c. lamp current too high
    d. a and b, above
    e. a, b, and c, above

27. Which of the following is a(are) possible cause(s) for the readout of an atomic absorption spectrophotometer being noisy with its flame on?
    a. burner head corroded
    b. nebulizer misadjusted
    c. lamp current misadjusted
    d. a and b, above
    e. a, b, and c, above

28. True or False? A misaligned lamp may cause an atomic absorption spectrophotometer to lose sensitivity.

29. Performance audits should be conducted for which of the following activities of the lead reference method?
    a. flow rate calibrations of high volume samplers
    b. lead analysis
    c. data processing
    d. a and b, above
    e. a, b, and c, above

30. During a performance audit of a high volume sampler, the sampler should be operated at _____(?)_____.
    a. 40 cfm
    b. 50 cfm
    c. 60 cfm
    d. its normal sampling flow rate

31. A high volume sampler should be recalibrated if a performance audit results in a relative difference between the sampler's measured flow rate and the audit flow rate of greater than ± _____(?)_____ percent.
    a. 3
    b. 7
    c. 15
    d. 20

32. Which of the following is(are) required in the performance auditing of lead analysis?
    a. Audit samples must consist of lead solutions deposited on glass-fiber filter strips.
    b. Audit samples must be prepared using reagents different from those used to generate lead calibration curves.
    c. Routine analysts must know the audit values before the audit.
    d. a and b, above
    e. a, b, and c, above

33. True or False? In auditing lead data processing, when the original calculations do not agree with the audit calculations, all original calculations for the period under audit should be recalculated.

34. Control charts should be maintained for _____(?)_____ audit results.
    a. high volume sampler flow-rate calibration
    b. lead analysis
    c. both a and b, above

35. Which of the following should be determined during a systems audit of a lead-monitoring network?
    a. types of high volume samplers and particulate matter filters used
    b. high volume sampler and atomic absorption spectrophotometer calibration procedures
    c. whether there is sufficient sampling and analysis documentation
    d. b and c, above
    e. a, b, and c, above

Section 7
Review Exercise Answers

Section 8

Automated Analyzers for Nonmethane Organic Compounds

Reading Assignment

Pages 1 through 31 of EPA 600/4-81-015 *Technical Assistance Document for the Calibration and Operation of Automated Non-Methane Organic Compound Analyzers.*

Reading Assignment Topics

- Reference measurement principle for nonmethane hydrocarbons
- Problems associated with analyzers for nonmethane organic compounds (NMOC)
- Installation and operation of NMOC analyzers
- Maintenance and troubleshooting of NMOC analyzers
- Calibration of NMOC analyzers
- Quality control for NMOC analyzers

Learning Goal and Objectives

Learning Goal

The purpose of this section is to familiarize you with automated nonmethane organic compounds (NMOC) analyzers that use the reference measurement principle for nonmethane hydrocarbons.

Learning Objectives

At the end of this section, you should be able to—
1. describe the nonmethane hydrocarbons reference measurement principle,
2. define and recognize the rationale for the term "nonmethane organic compounds,"
3. express NMOC concentrations in appropriate reporting units,
4. recognize and describe solutions for at least eight potential problems associated with NMOC analyzers, sample handling systems, or analyzer shelters,
5. describe at least eight activities involved in the installation of NMOC analyzers,

6. recognize at least six items that should be included in an analyzer logbook, and

7. describe maintenance, troubleshooting, calibration, and quality control procedures for NMOC analyzers.

Reading Guidance

- Although analyzers using the reference measurement principle for nonmethane hydrocarbons, which is described in the second paragraph of page 3 of the reading assignment, are available, none of them have been designated as reference methods by the U.S. EPA.

- The traceability requirement described on page 25 of the reading assignment should be applied where possible to all gaseous standards.

- Refer often to the tables and figures of the assigned reading material as you progress through the assignment.

- When you have finished the reading assignment, complete the review exercise for Section 8. It begins on the following page.

- After you have answered the review exercise questions, check your answers. The correct answers are listed on the page immediately following the review exercise.

- For any review exercise questions that you answered incorrectly, review the page(s) of the reading assignment indicated on the answers page.

- After you have reviewed your incorrect answers (if any), take the **Final Examination** for this course. Follow the directions listed in the Course Introduction section of this guidebook.

- Your course grade results will be mailed to you.

Review Exercise

Now that you've completed the assignment for Section 8, please answer the following questions. These will help you determine whether you are mastering the material.

1. The reference measurement principle for nonmethane hydrocarbons consists of separating methane from other organic compounds by ____(?)____ and measuring methane and other organic compounds by ____(?)____.
   a. thin layer chromatography, flame ionization detection
   b. liquid chromatography, ultraviolet photometry
   c. gas chromatography, flame ionization detection
   d. ion chromatography, infrared spectroscopy

2. Which of the following parameters are measured directly by analyzers using the nonmethane hydrocarbon reference measurement principle?
   a. methane
   b. total hydrocarbons
   c. nonmethane hydrocarbons
   d. a and b, above
   e. a, b, and c, above

3. True or False? A flame ionization detector responds uniformly from one hydrocarbon species to another.

4. Which of the following enhance(s) the uniformity of response of a flame ionization detector to different hydrocarbons?
   a. injection of hydrocarbons into detector via an air carrier
   b. injection of hydrocarbons into detector via an inert carrier gas
   c. both a and b, above

5. The concentration resulting from the subtraction of methane concentration from total hydrocarbon concentration, determined by flame ionization detection, should be referred to as nonmethane organic compounds (NMOC) concentration because ____(?)____.
   a. it includes oxygenated organics that contribute to the increase of photochemical oxidant concentrations
   b. flame ionization detectors exhibit some sensitivity to oxygenated compounds
   c. both a and b, above

6. Hydrocarbon concentrations should be reported as ppmC in order to
   ____(?)____.
   a. eliminate the confusion of using several different concentration units
   b. normalize the responses of flame ionization detectors to different organic compounds
   c. both a and b, above

7. Six ppm of propane (C_3H_8) is equal to ____(?)____ ppmC.
   a. 6
   b. 2
   c. 9
   d. 18

For each of questions 8 through 10, match the NMOC analyzer problem with its appropriate remedy.

8. large differences in the per-carbon response to different NMOC species

9. variations in oxygen concentration affect sensitivity of flame ionization detector

10. NMOC concentrations are subject to large, relative errors because they are the differences of relatively large and nearly equal numbers

a. all analyzer channels (total organic compounds, methane, NMOC) should be properly calibrated

b. NMOC response should be calibrated to a propane standard

c. calibration gases should be diluted to ensure that they contain $20.9 \pm 0.3\%$ oxygen

11. Temperature inside the monitoring shelter of an NMOC analyzer should be maintained within a range of ____(?)____°C.
    a. 10 to 40
    b. 20 to 30
    c. 20 to 40
    d. 30 to 50

12. Which of the following can prevent moisture in sample air from damaging NMOC analyzers?
    a. sample line moisture traps
    b. maintaining temperature of monitoring shelter within 5 to 7°C of outside temperature
    c. both a and b, above

13. Voltage supplied to an NMOC analyzer should be monitored ____(?)____.
    a. when the analyzer is installed
    b. at the beginning of heating and cooling seasons
    c. both a and b, above

14. Which of the following should be complied with when installing a sample-air manifold for an NMOC analyzer?
    a. air conditioning vents should be positioned away from the manifold
    b. the manifold's sample ports should be pointed toward the floor of the shelter
    c. both a and b, above

15. The sample manifold of an NMOC analyzer should be regularly inspected for ____(?)____.
    a. particulate matter
    b. moisture
    c. leaks
    d. a and c, above
    e. a, b, and c, above

16. True or False? An NMOC analyzer should be set up and operated immediately after it is unpacked.

17. True or False? Only clean or new Teflon® tubing should be used to supply hydrogen to an NMOC analyzer.

18. Hydrogen used by NMOC analyzers should contain less than ____(?)____ ppmC total organic compounds (TOC).
    a. 0.1
    b. 1
    c. 5
    d. 10

19. True or False? Signal cable used to connect an NMOC analyzer to its concentration readout device should not be spliced and should be as long as possible.

20. Sample-gas tubing for an NMOC analyzer should be composed of ____(?)____.
    a. polypropylene
    b. polyvinyl chloride
    c. Teflon®
    d. both b and c, above

21. True or False? An all-Teflon® particulate matter filter should be installed in the sampling line of an NMOC analyzer.

22. Which of the following should be accomplished before operating an NMOC analyzer?
    a. understand the basics of analyzer operation
    b. determine that there are no leaks in the analyzer's hydrogen-flow system
    c. prepare a set of analyzer operating summary sheets
    d. a and b, above
    e. a, b, and c, above

23. An NMOC analyzer logbook should contain which of the following?
    a. analyzer's instruction manual
    b. analyzer identification
    c. calibration, routine checks, and maintenance data
    d. b and c, above
    e. a, b, and c, above

24. Multipoint calibrations of NMOC analyzers should be ____(?)____.
    a. performed once per week
    b. performed once per month
    c. performed once per year
    d. based on analyzer performance

25. Which of the following operational parameters of an NMOC analyzer should
    be routinely checked?
    a. pressure gauge readings
    b. rotameter readings
    c. pyrometer readings
    d. a and b, above
    e. a, b, and c, above

26. Which of the following maintenance activities for an NMOC analyzer should
    be performed at least once every 180 days?
    a. leak check of hydrogen flow systems
    b. head pressure check of sample pump
    c. soiling check of sample line particulate matter filter
    d. a and b, above
    e. a, b, and c, above

27. Which of the following operating parameters of an NMOC analyzer can be
    evaluated from a chromatogram?
    a. sensitivity
    b. gate timing
    c. both a and b, above

28. Which of the following indicate(s) possible leaks or obstructions in the flow
    systems of an NMOC analyzer?
    a. decreased pressure gauge readings of flow systems
    b. calibration curve drift in one direction
    c. inability to perform balance procedures
    d. a and b, above
    e. a, b, and c, above

29. True or False? A leak in a gas-flow system of an NMOC analyzer should be
    identified with leak detector solution only after the general area of the flow
    system containing the leak has been determined by pressurizing the flow
    system.

For each of questions 30 through 32, match the NMOC analyzer response with its appropriate calibration standard when obtaining NMOC data for predicting ozone concentrations.

30. Methane (CH_4)
31. Total Organic Compounds (TOC)
32. NMOC

a. methane
b. ethane
c. propane

33. Zero air used in the calibration of NMOC analyzers should contain which of the following?
    a. 20.9 ± 0.3% oxygen
    b. nitrogen
    c. carbon monoxide
    d. a and b, above
    e. a, b, and c, above

34. Zero air used in the calibration of NMOC analyzers should contain less than _____(?)_____ ppmC.
    a. 0.1
    b. 1
    c. 2
    d. 5

35. True or False? Air used in the preparation of zero air for the calibration of an NMOC analyzer should be obtained from air inside the analyzer's monitoring shelter.

36. True or False? A heatless air dryer should be used to remove moisture from zero air used in the calibration of NMOC analyzers.

37. Calibration gases must be delivered to the inlet of an NMOC analyzer _____(?)_____ atmospheric pressure.
    a. at
    b. below
    c. above

38. Calibration gases used to calibrate an NMOC analyzer should be traceable to a _____(?)_____.
    a. standard reference material of the National Bureau of Standards
    b. certified reference material
    c. either a or b, above

39. Methane and propane standards used to calibrate NMOC analyzers should be analyzed _____(?)_____ to ensure that concentrations of organic contaminants remain less than 0.1 ppmC.
    a. weekly
    b. monthly
    c. quarterly
    d. yearly

40. Which of the following must be complied with when calibrating an NMOC analyzer?
    a. calibration manifold vented to the atmosphere
    b. calibration gases introduced through analyzer's calibration port
    c. gas flow to calibration manifold 20 to 50% greater than analyzer's sample flow demand
    d. a and c, above
    e. a, b, and c, above

41. For dynamic dilution calibrations of NMOC analyzers, diluent-to-pollutant flow rate ratios should be greater than _____(?)_____:1.
    a. 2
    b. 10
    c. 50
    d. 100

42. True or False? Preparing a calibration gas by dynamic dilution requires accurate flow rate measurements.

43. When using a soap bubble flowmeter, select a bubble tower that will permit a flow measurement for at least _____(?)_____ seconds.
    a. 5
    b. 10
    c. 15
    d. 30

44. Wet-test meters should generally be used to measure flows within a range of _____(?)_____ meter revolution(s) per minute.
    a. ½ to 1
    b. ½ to 2
    c. ⅓ to 3
    d. 1 to 3

45. True or False? Flows through wet-test meters should be measured by complete meter revolutions only.

46. Under the conditions described below, the NMOC concentration of an air sample obtained from an NMOC analyzer is _____(?)_____ ppmC.
    a. 0.100
    b. 0.300
    c. 0.900
    d. 1.17

    Given: Methane calibration curves:
                 CH_4 response: 9.000X + 0.200
                 TOC response: 9.900X + 0.100
              Propane calibration curve:
                 TOC response: 3.300
              NMOC analyzer responses to air sample:
                 CH_4 response: 6.5% of analyzer's concentration range
                 TOC response: 10.0% of analyzer's concentration range

47. Under the conditions described below, the span drift of the NMOC analyzer is
    ____(?)____ percent.
    a. 0.0
    b. 9.9
    c. 10.0
    d. 10.1
        Given: Analyzer's response to Level 1 span: 88% of analyzer's full scale
                  Analyzer's response to Level 1 zero: 0% of analyzer's full scale
                  Level 1 span concentration: 80% of analyzer's full scale
                  Slope of analyzer's multipoint calibration curve: 1.100

48. True or False? Level 2 zero and span gas must be introduced through the nor-
    mal sample inlet of an NMOC analyzer.

Section 8
Review Exercise Answers

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

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16. ABSTRACT

This guidebook was developed for use in the Air Pollution Training Institute's Correspondence Course 438, "Reference and Equivalent Measurement Methods for Ambient Air Monitoring." It contains reading assignments and review exercises covering the following topics:
    - Reference Method for Sulfur Dioxide
    - Automated Equivalent Measurement Methods for Sulfur Dioxide
    - Reference Methods for Ozone
    - Reference Method for Total Suspended Particulate Matter
    - Reference Methods for Carbon Monoxide
    - Reference Methods for Nitrogen Dioxide
    - Reference Method for Lead
    - Automated Analyzers for Nonmethane Organic Compounds
 	
    This guidebook is designed for use in conjunction with "APTI Course 464 Analytical Methods for Air Quality Standards: Student Manual" (EPA 450/2-81-018b) and "Technical Assistance Document for the Calibration and Operation of Automated Non-Methane Organic Compound Analyzers" (EPA 600/4-81-015).

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