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INSTRUCTOR MANUAL
INDUSTRIAL HYGIENE CHEMISTRY COURSE

LESSON NUMBER 11

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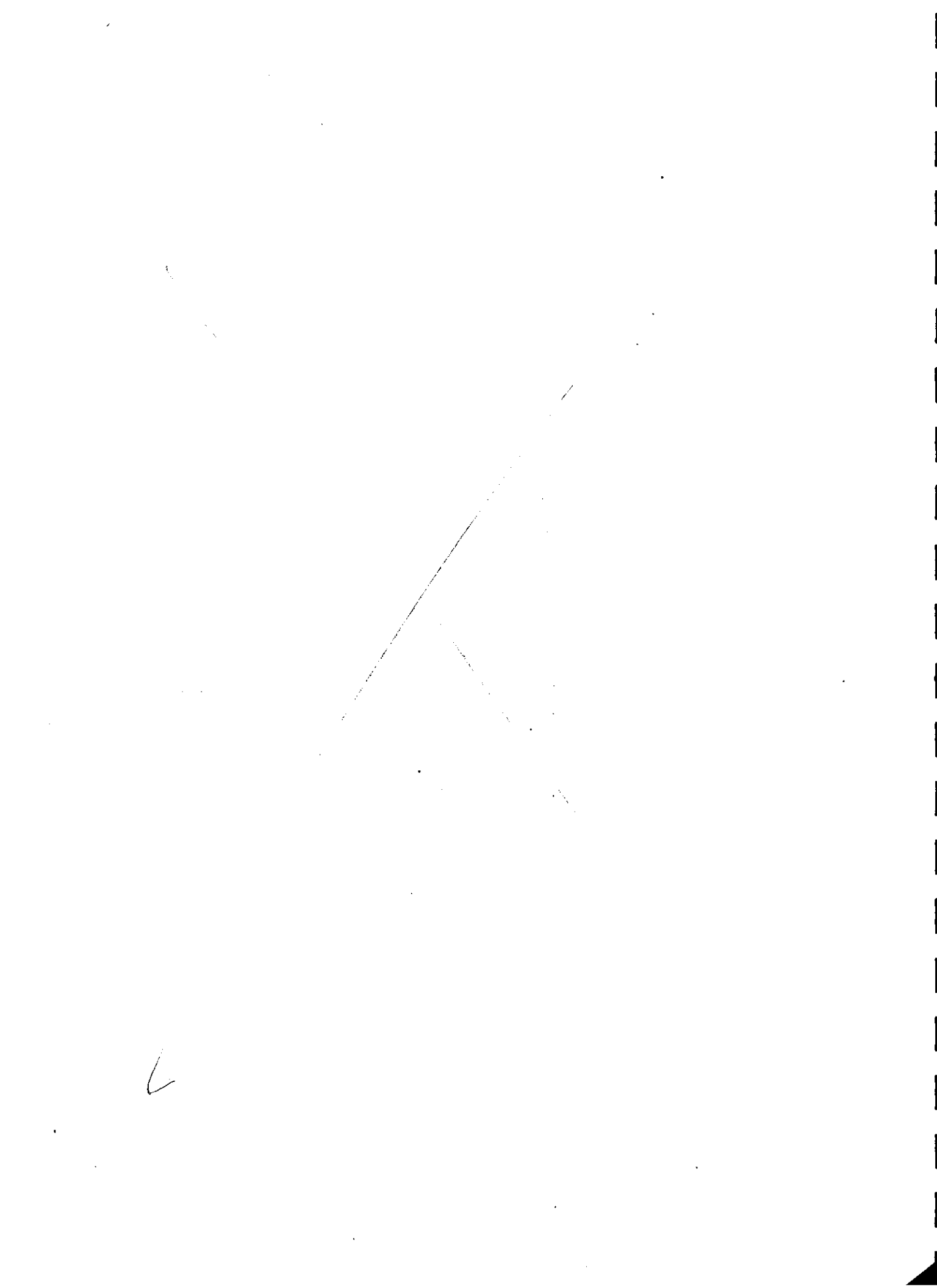

INTRODUCTION

This Instructor Manual has been prepared for industrial hygienists and analytical chemists participating in the National Institute for Occupational Safety and Health's Regional Training Program. The purpose of this Manual is to assist these professionally qualified, but possibly inexperienced, instructors in the preparation and conduct of a one-week "Industrial Hygiene Chemistry" course. This Manual will guide instructors through both lecture and laboratory lessons. It is complemented by a matching Student Manual. The course is recommended for students having, as a minimum, an undergraduate degree in chemistry (or its equivalent) along with at least one year's experience in instrumental analysis.

It is not necessary for instructors to have had prior teaching experience although such experience would be desirable. All instructors should be thoroughly familiar with industrial hygiene chemistry procedures, instruments and equipment relevant to the subject areas they will teach. In addition, each instructor should attend the course director's orientation seminar presented before the start of each one-week "Industrial Hygiene Chemistry" course.

The remainder of this introduction describes the course objectives, lessons, and the organization and format of the documentation in each lesson, including lecture and laboratory lesson plans.

Course Objectives

The following course objectives will be attained by graduates of this program:

- Given a particular chemical health hazard commonly found in the occupational environment, the trainee will be able to select an appropriate sampling strategy using available sampling techniques and to select a corresponding appropriate analytical method for quantitative characterization of the sample by using his knowledge gained from the course and technical information referenced in the course.

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- Trainee will be able to apply his knowledge of wet chemical and/or instrumental analysis in employment of current methodologies for evaluating the typical work environment.

- Trainee will be able to perform and evaluate quantitative analytical determinations for four classes (types) of hazardous substances using a correspondingly different method for each class or type.

- Given the analytical results obtained through proper measurement procedures, the trainee will be able to define the data in terms of actual environmental concentration levels and to interpret the results in light of existing exposure standards.

Lessons

18 lessons are presented in this course:

- Introduction to Course
- Introductory Topics
- Direct Reading Instruments
- Air Flow Calibration and Sampling
- Ion Selective Electrode Laboratory
- Introduction to Spectrophotometry
- Instrumentation and Application of Spectrophotometry
- Colorimetric Determination of Free Silica (Quartz) Laboratory
- Introduction to Spectroscopy
- Atomic Absorption Spectrometry
- Atomic Absorption Spectrometry Laboratory
- Introduction to Chromatography
- Insturmentation and Application of Chromatography
- Gas Chromatography of Organic Solvents Laboratory
- Titrametric Determination of SO_2 Laboratory
- Colorimetric Determination of SO_2 Laboratory
- Biological Monitoring
- Related Topics

Lectures

Each lesson that is to be presented as a lecture is documented in a standardized format.

A. Lecture Cover Sheet

A cover sheet for each lecture presents the following information:

- Lesson title
- Lesson number and length
- Behavioral objective
- Scope of the lesson
- List of visuals
- List of exhibits
- List of equipment needed for the lesson

B. References

After the cover sheet, there is a list of references. These references are keyed to the paragraphs within each lesson. The number in parenthesis following each paragraph is the reference number. These references are included so that the instructor, if he wishes, may further research specific instructional subject matter.

C. Additional Readings

Following the reference list, in most lessons, is another listing called "Additional Reading." This bibliography contains books and articles which are generally pertinent to the subject area covered in this lesson. These are considered as important secondary reference sources.

D. Expanded Outline (left-hand page)

On the left-hand page, beginning after the Additional Readings section, is an expanded outline. This outline indicates the information that should be emphasized and covered during the lecture. The sequence of the outline should be followed during

teaching. The expanded outline gives sufficient information to explain the brief outline which is on the right-hand page. All test questions (both self tests and course evaluator) come from the expanded outline. Additionally, there are descriptions of the visuals within the outline.

E. Brief Outline (right-hand page)

This page consists of a notes column and the outline.

1. Notes Column - times (both elapsed and projected) are indicated in this column. The elapsed time designates the time it should take the instructor to reach this point in the lecture starting from 0 at the beginning of each lecture. The elapsed time is in parentheses. The projected time designates the time it should take the instructor to reach the next major portion of the outline. A major portion of an outline is designated by a capital letter in the outline. In addition, transitional phrases connecting the major outline portions are included in the notes column. These phrases are to assist the instructor in bridging from one section of the outline to the next. Notations of what visual, exercise, table, etc., should be introduced at a given point in a lesson and miscellaneous notes to the instructor are contained also in this column.

2. Outline - this is a brief outline corresponding to the expanded outline on the facing page. Words and phrases in the brief outline key the instructor to the lesson's subject content and to the expanded outline on the left-hand page. There is sufficient space between the key words in the brief outline for the instructor to write his own additional notes when he is preparing his lecture.

F. Exercises and Problems

In some lessons, exercises and problems are included. These are given during class time. The answers to the problems are worked out with students after they have had an initial try at completing them on their own. Answers are provided in the Instructor Manual.

G. Self Tests

Self tests are included after most lessons. The Instructor Manual contains the correct answers, whereas the Student Manual does not. The students should first answer the questions, and then the instructor should review the answers, explaining fully the reasons for the correct answers. The self tests are not scored by the instructor and no records are kept of the individual student's performance. The instructor should use the information from the discussion of self tests to remove student misunderstandings or lack of understanding.

H. Copies of Visuals

Copies of visuals that are to be shown in a lecture are included at the end of that lesson documentation. These can be useful in preparing for the lecture presentation.

I. Homework

No specific homework assignments are included within the lesson documentation. However, there is a great quantity of information for the students to absorb during this one-week course. Therefore, students should be urged to review nightly all lessons covered during the day and all lessons to be presented on the following day. In particular, they should become familiar with the laboratory procedures for the following day. There is much to be accomplished in every laboratory and little time to do it. If the students are familiar with the procedure, the laboratory experiments will progress much more smoothly.

Laboratories

Each lesson that is to be presented as a laboratory is documented in standardized format consisting of four elements.

A. Laboratory Cover Sheet

A cover sheet for each laboratory presents the following information:

- Lesson title
- Lesson number and length
- Behavioral objective
- Scope of the lesson
- List of equipment, apparatus and forms

B. Special Preparation Section

This section will follow the laboratory cover sheet, and includes specific directions that must be followed prior to actual class time. These instructions are concerned with the preparation of apparatus, facilities, chemicals and materials that are necessary for the laboratory session.

C. Laboratory Procedures

The procedures for performing each laboratory are fully documented on the left-hand page. The elapsed and projected times are indicated for some lessons with the elapsed times appearing in parentheses. The right-hand page is a blank page for notes on specifics of the laboratory to aid the individual instructor in giving an efficient lesson.

D. Figures and Forms

Equipment figures and student forms are included after the procedures. The figures are presented to aid the instructor in setting up the experimental equipment. The forms are to be used by the students during the laboratory to assist them in recording, calculating and analyzing data.

LESSON TITLE	LESSON NUMBER	LESSON LENGTH
Atomic Absorption Spectrometry Laboratory	11	4:00

BEHAVIORAL OBJECTIVE

The student will be able to calibrate and operate an atomatic absorption spectrophotometer for the purpose of quantitating the amounts of metals (lead in this case) collected on filters such as cellulose membranes.

SCOPE

Preparation of aqueous calibration standards for lead
Collection of particulate lead on a cellulose membrane filter
Preparation of samples for analysis
Optimization of atomic absorption spectrophotometer operating parameters for lead
Data collection on standards and samples
Calculation of results and interpretation

EQUIPMENT, APPARATUS, AND FORMS

Equipment and chemicals for each section of the lab is as follows:

B. Hot Plates
10-ml. volumetric flasks (borosilicate glass)
100-ml. volumetric flasks (borosilicate glass)
125-ml. Phillips beakers (borosilicate glass)
1, 2, 5, 10 and 25 ml. pipettes (borosilicate glass)
10-ml. graduated cylinders (glass or polyethylene)
Detergent solution
Concentrated HNO_3
Lead-free water

C. Tweezers
0.45 - μm. cellulose membrane filters
Filter cassettes
Rotameters, 0-5 liters per minute (and calibration curves)
Vacuum pumps such as the personal sampling pumps
Flexible tubing
Thermometer

EQUIPMENT, APPARATUS, AND FORMS (continued)

Stopwatch
125-ml. Phillips beaker cleaned as described in Section B

D. 100-ml. volumetric flasks cleaned as described in Section B
1, 2, 5, 10 and 25-ml. pipettes cleaned as described in Section B
1000 μg./ml. Lead Standard
1% (v/v) HNO_3

E. Hot plates
Tweezers
125-ml. Phillips beakers cleaned as described in Section B
Reference sample
Filter blank
Concentrated redistilled HNO_3
10-ml. volumetric flasks cleaned as described in Section B
Lead-free water

F. Atomic absorption spectrophotometer with digital readout or a recorder
Lead hollow cathode lamp

G. Same as F

Forms:

Collection of Lead in an Air Sample
Location of Optimum Observation Heights in Flame
Measurement of Standards and Samples

SPECIAL PREPARATION

Special preparation for each section of the laboratory should be noted by the instructor before the lab begins, and concurrently as the experiments progress.

General:

It is preferrable to have several atomic absorption instruments available for the students. This will allow them opportunity to handle all equipment and perform, personally, all parts of the laboratory. It is recognized that this may not be feasible in many situations. A second method is staggering the starting and finishing times for each student. This may also be impractical. In those cases, the students will be required to work in teams.

Section:

B. Washing glassware is an essential step for successful trace analysis and should be done by each student. Large amounts of lead-free water will be required for rinsing. This can be prepared by double distillation or ion exchange methods and stored in polyethylene carboys. Occasionally, regular distilled water is lead-free. This can be checked by the instructors in advance by analyzing representative samples of their distilled water which has been concentrated by a factor of 5 through evaporation. If no lead can be detected in the concentrate, it should be below 0.02 μg./ml. which is acceptably low.

C. If apparatus is limited, students may work in teams to collect a few air particulate samples. They should become familiar with the procedure. The flow rate should be checked periodically during collection.

D. Aqueous lead standards can be synthesized while the lead-air sample is being collected. The 1000 μg./ml. lead standard can be prepared by dissolving 1.598 g. of $Pb(NO_3)_2$ in a 1 liter volumetric flask using 10% (v/v) HNO_3. Alternatively, it can be purchased from any of a number of chemical supply houses. The 1% (v/v) HNO_3 should be prepared in bulk using redistilled HNO_3 lead-free water.

E. Reference samples should be prepared in advance by spotting 0.10 ml. of 1000 μg./ml. lead standard onto individual filters. If micropipettes are used, each filter should contain 100 ± 2 μg. of lead. The solution

is dried and the filters stored in a dry, dust-free environment. The lead hollow cathode lamps should be turned on at the start of this section as they will be ready for use in the following section. Normally, the solid portion of a cellulose membrane filter is dissolved by hot concentrated HNO_3 in 2-5 minutes. A yellow color due to incompletely oxidized organic matter is of no consequence in the flame excitation process. In the rare case where fibers or particles remain after the first digestion, a second portion of concentrated HNO_3 may be needed.

F. With some instruments, the 25 µg./ml. standard may show some negative deviation from Beer's Law in which case the upper portion of the calibration should be drawn as a curve.

TITLE	LESSON NUMBER
Atomic Absorption Spectrometry	11

A. Introduction and Review (---) 0:20

1. Describe the seven parts of the laboratory.

   - Cleaning of glassware
   - Collection of lead in an air sample
   - Synthesis of aqueous lead standards
   - Preparation of samples for analysis
   - Set up operating conditions for atomic absorption
   - Measure standards and samples
   - Compare results

2. Review briefly laboratory manipulations and concentration units.

B. Cleaning of Glassware (0:20) 0:30

1. Clean all glassware by washing in detergent followed by tap water and distilled water rinse.

2. Soak volumetric flasks and pipettes in 1:1 HNO_3 for 3 minutes; rinse with distilled water and finally with Pb-free water. The latter may be prepared by double distillation or ion exchange methods and stored in polyethylene carboys.

3. Transfer about 3 ml of concentrated HNO_3 with the 10-ml. graduate to each of three 125-ml. Phillips beakers. Heat on a hot plate (in the hood)

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until acid condenses and runs down the sides. Discard the acid into a waste bottle (in the hood), rinse with distilled water and finally with Pb-free water.

C. Collection of Lead in an Air Sample (0:50) 0:40

1. Using clean tweezers, place a 0.45-μm. cellulose membrane filter into a filter cassette.

2. Attach the exit end of the filter cassette via a short tube or special adapter to a rotameter capable of monitoring an air flow of 2 liters per minute.

3. Connect the rotameter to a vacuum pump with a short length of flexible tubing.

4. Place the assembly in a suitable location such as near a well-traveled road or even in an adjacent area of the building.

5. Start the pump and adjust the flow rate to 2 liters per minute. Immediately start the stopwatch.

6. Record the barometric pressure and temperature in the sampling location.

7. Pass 100 liters of air through the filter.

8. At the appropriate time, stop the collection, transfer the filter from the cassette to a 125-ml. Phillips beaker using tweezers and reserve for section E.

D. Synthesis of Aqueous Lead Standards (1:30) 0:30

1. Using the acid-cleaned pipettes and volumetric flasks, pipette 10.0 ml. of 1000 μg./ml. lead stock solution into a 100-ml. volumetric flask and

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dilute (with thorough mixing) to the mark with 1% (v/v) HNO_3. This solution contains 100 µg./ml. of lead.

2. Into five separate 100-ml. volumetric flasks, pipette 1.0, 2.0, 5.0, 10.0 and 25.0 ml. of 100 µg./ml. lead solution from (1) above. Make each flask to volume with 1% (v/v) HNO_3. These standards contain 1.0, 2.0, 5.0, 10.0 and 25.0 µg./ml. of lead, respectively.

E. Preparation of Samples For Analysis (2:00) 0:30

1. Secure from the instructor a reference sample on a cellulose membrane filter and also an unused filter to serve as a blank.

2. Place each filter (use tweezers to handle) in separate Phillips beakers.

3. Add about 3 ml. of concentrated redistilled HNO_3 to each of these beakers and to the beaker containing the lead-air sample (Section C).

4. Carefully heat on a hot plate in a hood. Avoid splattering. In 2-5 minutes a clear yellow solution will result. Continue heating until the acid volume is about 1-ml. or less but do not take to dryness.

5. Quantitatively transfer each sample to a 10-ml. volumetric flask with the aid of lead-free water. Dilute to the mark with thorough mixing.

F. Establish Operating Conditions for the Spectrometer (2:30) 0:40

1. The lead hollow cathode lamp will be ready for use.

2. With the aid of the instructor, light the air-acetylene flame and adjust to oxidizing conditions, i.e., fuel lean so the color is blue (non-luminous).

3. Aspirate the 25 µg./ml. standard and adjust the wavelength setting at 283.3 nm. to give maximum absorbance. The monochromator is now

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properly set for the lead line.

4. With the burner height adjusting knob, raise the burner until the light beam just passes over the base of the flame. Use lead-free water to zero the instrument and then record the absorbance of the 10 µg. /ml. lead standard. Lower the burner in measured increments (at least 5 steps) and record the absorbance corresponding to each height in the flame.

5. Plot the absorbance against the height of observation in the flame and set the instrument at the indicated height for maximum absorbance.

G. Measurement of Standards and Samples (3:10) 0:30

1. Record in random order and in duplicate, the absorbances for the five aqueous standards and the three acid-digested samples.

2. Plot the average absorbances vs. the concentrations for the five standards and draw the best fitting straight line.

3. Read from the curve the concentrations corresponding to the average absorbances for the blank and samples.

4. Multiply each value for the samples by 10 to obtain total µg. / filter.

5. Subtract the blank value from the reference sample and the air sample.

H. Comparison of Results (3:40) 0:20

1. Obtain the data of each student for the reference sample and the air sample.

2. The instructor will specify the correct value for the reference sample.

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3. Evaluate the precision and accuracy of the results on the reference sample.

4. Using the corrected air volume V_c from section C, calculate the μg. Pb per liter of air. This also corresponds to the concentration of Pb in mg./m.^{3}.

5. Discuss the range of values obtained for the air samples.

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Collection of Lead in An Air Sample

(Section C)

Name: _____

Date: _____

Flow Rate: _____ liters/minute

Time Start: _____

Time End: _____

Collection Time: _____ minutes

Total Volume Uncorrected (V): _____ liters

Temperature (T) _____ @C

Pressure (P): _____ mm. Hg

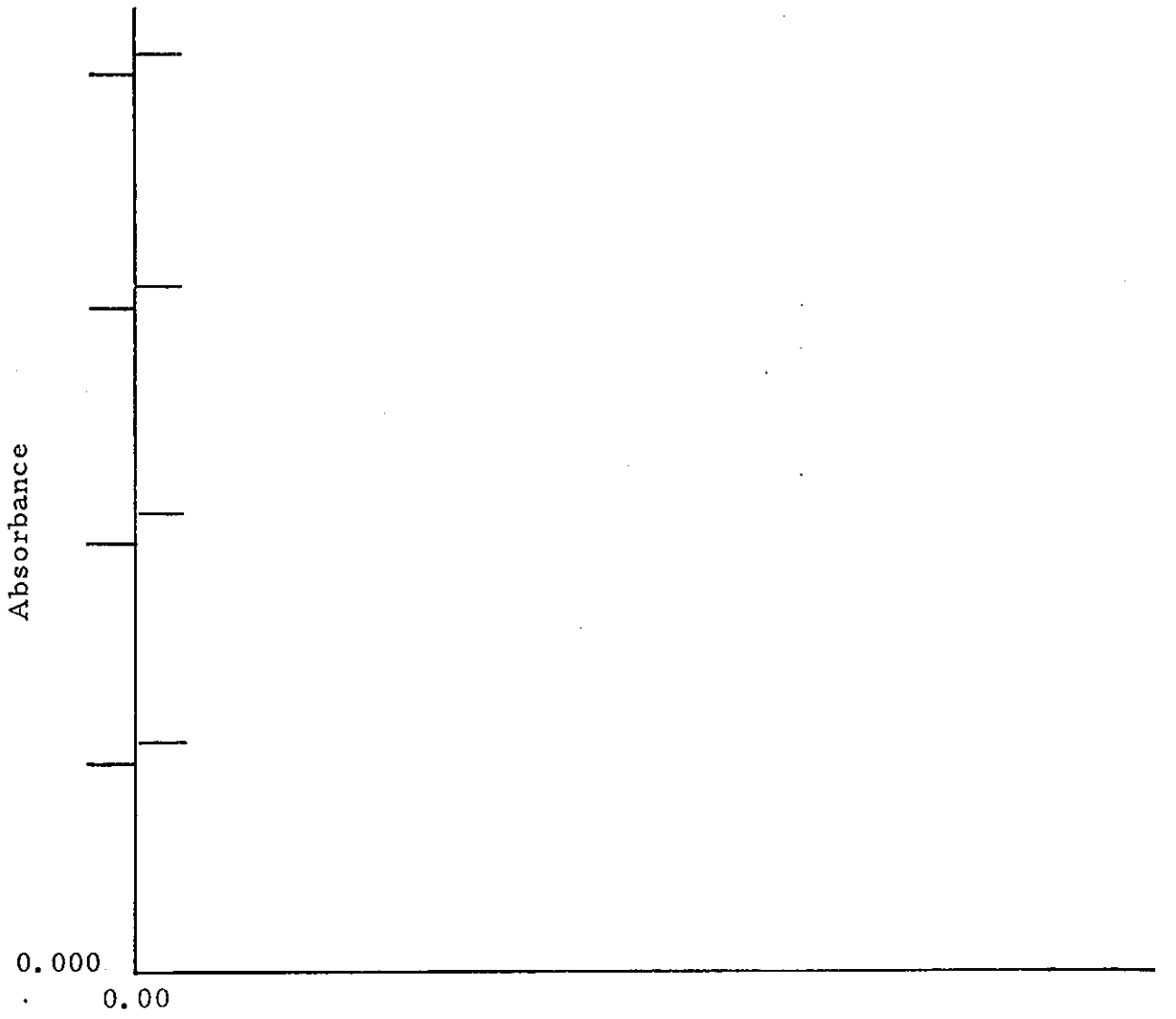
Corrected Volume, Vs, at 25° C and 760 mm. Hg is

$$Vc = V \times \frac{P}{760} \times \frac{298}{T + 273}$$

$$Vc = \_\_\_\_ \times \frac{\_\_\_\_}{760} \times \frac{298}{\_\_\_\_} = \_\_\_\_ \text{ liters}$$

Location of Optimum Observation Height in Flame
(Section F)

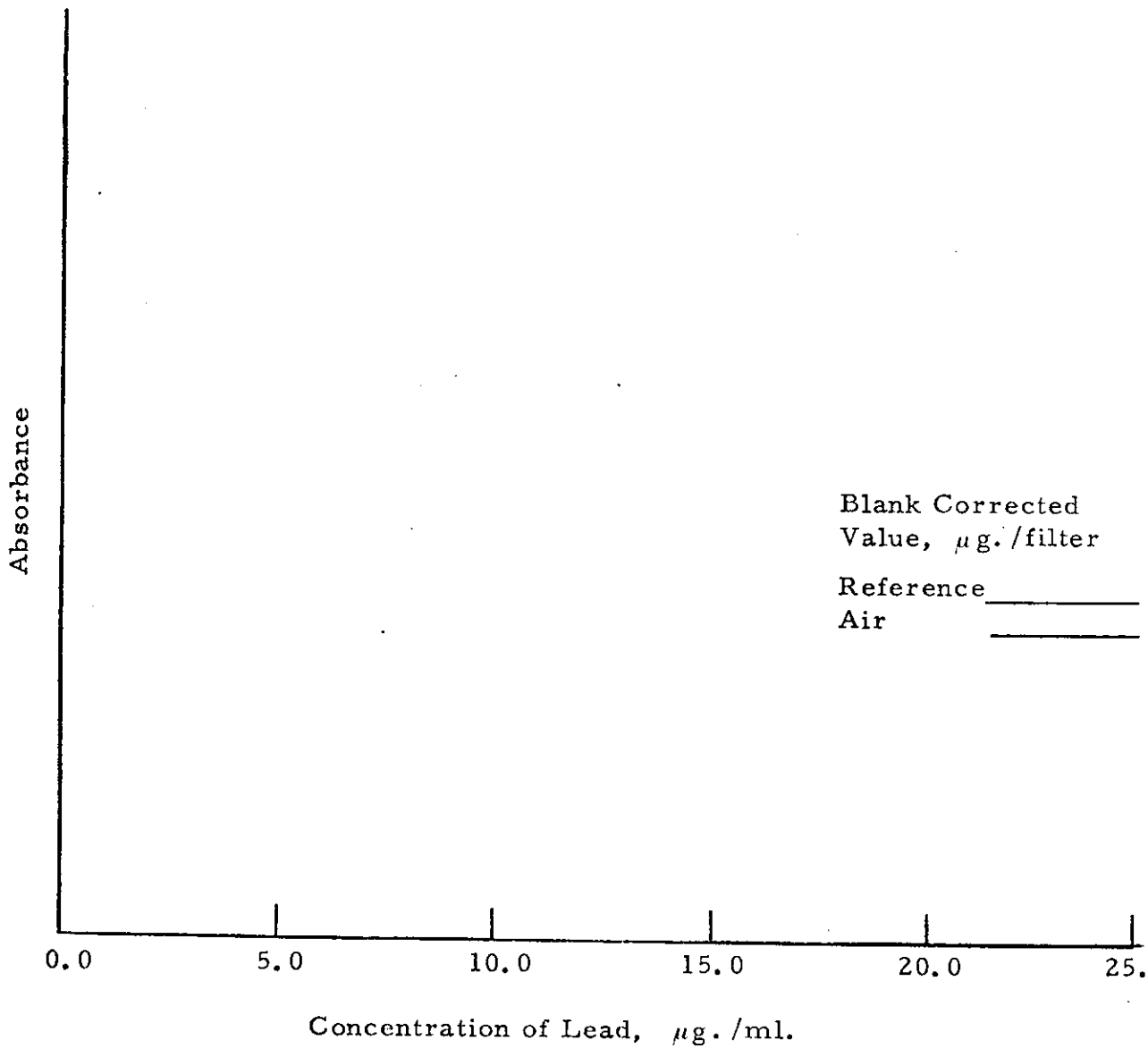
Position	Observation Height in Flame (cm.)	Absorbance of 10 µg./ml. Lead
Base	0.0	
1		
2		
3		
4		
5		



Measurement of Standards and Samples
(Section G)

Lead Concentrations (μg./ml.) Standards	Absorbances Rep. 1.	Rep. 2.	Ave.
1.0			
2.0			
5.0			
10.0			
25.0			

Samples	μg./ml.	μg./filter
Reference		
Air		
Blank		



Blank Corrected Value, μg./filter

Reference _____

Air _____