

ED75-2

PR82-147687

INSTRUCTOR MANUAL
INDUSTRIAL HYGIENE CHEMISTRY COURSE

LESSON NUMBER 14

April 1975

Prepared for:

National Institute for Occupational Safety and Health
Rockville, Maryland

Contract Number CDC-99-74-72

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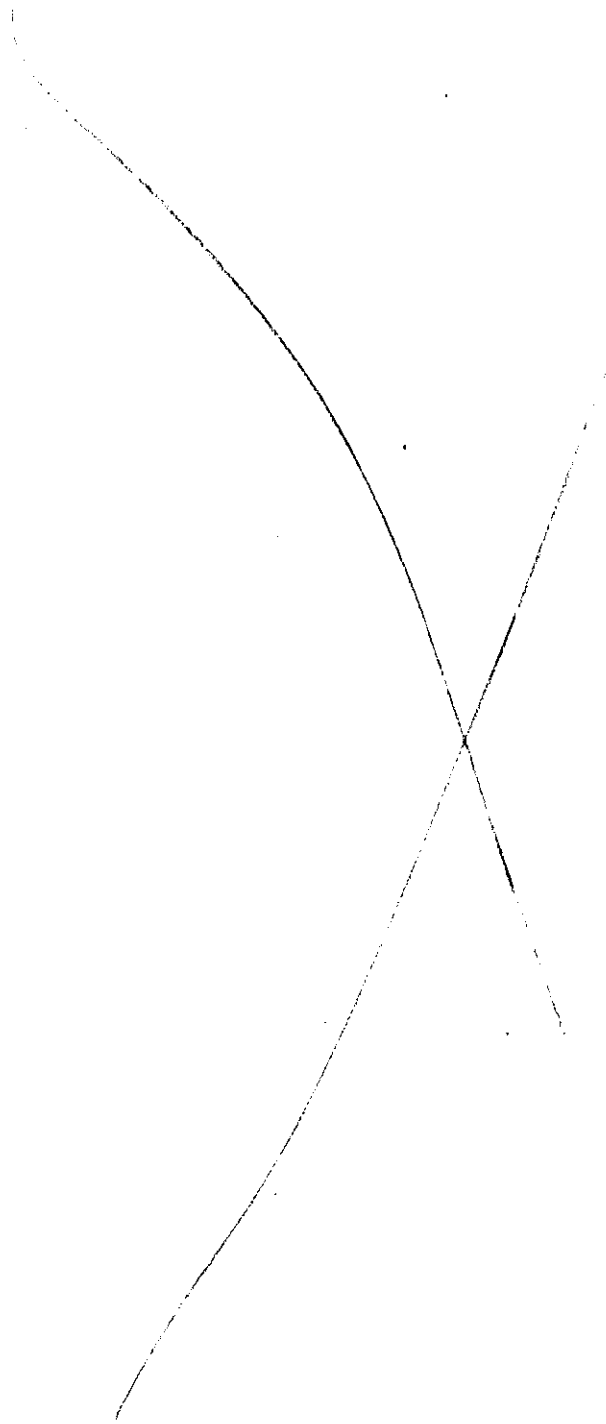
ACKNOWLEDGEMENTS

Dunlap and Associates, Inc., of Darien, Connecticut wishes to acknowledge with sincere appreciation the support received from the National Institute for Occupational Safety and Health in the administration and conduct of this project. In particular, we would like to express our gratitude to Dr. Thomas Purcell, the Project Officer.

We wish also to thank our consultants who contributed directly to the preparation of course materials: Mr. J.D. Johnson of Spectrogram Corporation, Dr. C.L. Grant of the Center for Industrial and Institutional Development - University of New Hampshire, Dr. Melvin W. First of the Harvard School of Public Health, and Mr. Adrian L. Lynch, private consultant.

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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.

Preceding page blank

- . 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
- . Instrumentation and Application of Chromatography
- . Gas Chromatography of Organic Solvents Laboratory
- . Titrametric Determination of SO₂ Laboratory
- . Colorimetric Determination of SO₂ 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
Gas Chromatography of Organic Solvents	14	4:00

BEHAVIORAL OBJECTIVE

The student will be able to operate and calibrate a gas chromatograph to quantitate benzene, chloroform, carbon tetrachloride and trichloroethylene adsorbed on charcoal in a glass tube.

SCOPE

Introduction
 Demonstration of a sampling train for organic solvents
 Learning to use a gas chromatograph
 Preparation of samples for desorption efficiency
 Preparation of standards
 Analysis of standards and samples
 Calculations and data analysis

EQUIPMENT, APPARATUS, AND FORMS

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

- B. Personal sampling Pump
 - Rotameter for use at 1 lpm
 - Charcoal sampling tubes with caps
 - Stopwatch
 - Thermometer
 - Manometer
 - Chromatoquality grade benzene, chloroform, carbon tetrachloride and trichloroethylene
- C. Gas chromatograph with flame ionization detector
 - Column (20 ft. x 1/8 in.) with 10% FFAP stationary phase on 80/100 mesh, acid-washed DMCS chromosorb W support.
 (Other columns can be used but will require somewhat different operating conditions.)
 - A digital integrator or strip chart recorder (planimeter optional)

EQUIPMENT, APPARATUS, AND FORMS (continued)

10- μ l. syringe
Spectroquality carbon disulfide
Demonstration mixture (see Special Preparation)
Bureau of Mines Grade A helium
Purified hydrogen
Filtered compressed air

- D. Parafilm sealed tubes containing known amounts of solvents adsorbed on charcoal
Parafilm sealed tube containing charcoal to use as a blank
Sampling tube for analysis
2.5-ml. graduated glass stoppered microcentrifuge tubes
0.50-ml. delivery pipets or 1.00-ml. graduated pipets
Files
Wires
10- μ l. syringes
Spectroquality CS₂
Mixture S
- E. 10-ml. volumetric flasks
1.00-ml. or 0.50-ml. graduated pipets or 500 μ l. syringes
5.00-ml. delivery pipets
Spectroquality CS₂
Mixture S
- F. Same as C except that the "Demonstration Standard" is replaced by the student standards and samples

Forms:

Sampling Organic Solvents
Sample Volumes - Desorption Experiments
Peak Areas For Standards and Samples
Calibration Curves for Organic Solvents
Calculations

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:

Cumulative time notations will not appear in the documentation of this laboratory. In fact, the sum of the several sections of the laboratory is greater than the four hours allocated. However, the laboratory can be completed within the allotted time by efficiently coordinating the working time of every student. Suggestions will be made in the following parts of the "Special Preparation" section. This will be dependent upon the number of students, quantities of equipment, and amount of work space. The instructors may decide that it is necessary for students to work in pairs.

Section:

- B. The charcoal adsorption tubes with caps are commercially available (Mine Safety Appliance Co., SKC, Inc. and others). A large batch made from the same lot of charcoal should be obtained. Since the desorption efficiency varies from one batch of charcoal to another, students can only compare results if all tubes are from the same lot of charcoal. In order to have a few extras, you should plan on seven tubes per student (they will be used in the desorption work).

The rotameter should be calibrated in advance of the laboratory.

A test chamber for sampling can be constructed and is desirable if possible. One type is described in L. D. White et al., Amer. Ind. Hyg. Assoc. J. 31, 225-232 (1970). Others are described in "Air Sampling Instruments For Evaluation of Atmospheric Contaminants," 4th Ed., Amer. Conf. of Gov. Ind. Hygienists, P.O. Box 1937, Cincinnati, Ohio, 45201 (1972). If a test chamber is used, the approximate concentration ranges most suitable for 10-liter samples are:

Benzene-----	10-30 ppm
Chloroform-----	30-60 ppm
Carbon Tetrachloride-----	10-20 ppm
Trichloroethylene -----	50-100 ppm

SPECIAL PREPARATION

- C. The conditions given are typical for the Perkin Elmer Model 900. The instructors will have to determine suitable conditions for other chromatographs. The instruments should be turned on well in advance of the laboratory so they will be at equilibrium when the laboratory starts.

Half of the students should learn to use the chromatographs while the other half prepare samples and standards. This will reduce congestion in the hood area. Further, each student should practice injecting a sample under the supervision of the instructor. The students who first prepare samples and standards can be taught to use the chromatographs as soon as their preparation is complete. Unfortunately, there will still be some waiting for access to the instruments. The amount will clearly depend on the number of students and the number of instruments. If this ratio is highly unfavorable, it may be necessary to have the students work in pairs.

The spectroquality CS_2 can be obtained from Matheson, Coleman and Bell or may be purified by the instructors.

The demonstration mixture is prepared by first mixing the four solvents (spectro or chromatoquality) to prepare an undiluted mixture. This is accomplished by transferring with a pipet to a glass stoppered flask the following quantities of each solvent:

	20.0 ml. carbon tetrachloride
	20.0 ml. benzene
Label This Mixture (S)	70.0 ml. trichloroethylene
	30.0 ml. chloroform

The demonstration mixture (comparable to the highest standard - D - in Section E) is prepared by pipetting 0.28 ml. of Mixture S into a 10-ml. volumetric flask which is made to volume using CS_2 .

- D. The most convenient way to prepare desorption tubes is to scribe with a file and break new 7-cm. adsorption tubes at the center of the urethane spacer between the two sections of graphite. The backup section is discarded. The remaining 5-cm. length is used for the desorption work by removing the urethane plug, pushing the charcoal down into the tube a bit with a 3-mm. diameter rod and covering the cut end with Parafilm. One tube per student (plus a few extras) are labeled Z and are set aside for use as blanks. The equivalent of two tubes per student (plus extras) are labeled Y. Into each of these tubes inject $7.0\mu\text{l.}$ of Mixture S directly into the charcoal using a microliter

SPECIAL PREPARATION

syringe. Reseal with more Parafilm. These samples must be prepared at least 12 hours in advance to insure complete adsorption onto the charcoal.

The unknown sampling tubes can also be prepared by injecting a known amount of the solvents (not necessarily in the same ratio as in Mixture S) onto a series of sampling tubes with the ends broken off. The ends would be capped with the plastic caps after the sample was applied.

Alternately, if a sampling chamber is available, the tubes can be prepared by drawing 10 liters of a known composition air through the tubes.

TITLE	LESSON NUMBER
Gas Chromatography of Organic Solvents	14
<p>A. Introduction</p> <p>1. <u>Precautions:</u></p> <ul style="list-style-type: none">Due to its toxicity all standard and sample preparation work involving the transfer of CS₂ must be conducted in a hood.The organic solvents being determined and the matrix (CS₂) are all volatile; and therefore, containers should be open to the atmosphere for the shortest possible time to avoid significant compositional changes. <p>2. Describe the six parts of the laboratory noting that after the sampling demonstration, half of the students should begin the sample and standards preparation while the other half learn to use the gas chromatographs:</p> <ul style="list-style-type: none">Demonstration of a sampling train for organic solventsLearning to use a gas chromatographPreparation of standardsAnalysis of standards and samplesCalculations and data analysis <p>B. Demonstration of a Sampling Train For Organic Solvents</p> <p>1. The sampling train consists of a personal sampling pump with an attached rotameter which has been calibrated with a representative charcoal sampling tube in the line.</p>	

- Due to its toxicity all standard and sample preparation work involving the transfer of CS₂ must be conducted in a hood.
- The organic solvents being determined and the matrix (CS₂) are all volatile; and therefore, containers should be open to the atmosphere for the shortest possible time to avoid significant compositional changes.

2. Describe the six parts of the laboratory noting that after the sampling demonstration, half of the students should begin the sample and standards preparation while the other half learn to use the gas chromatographs:

- Demonstration of a sampling train for organic solvents
- Learning to use a gas chromatograph
- Preparation of standards
- Analysis of standards and samples
- Calculations and data analysis

B. Demonstration of a Sampling Train For Organic Solvents

1. The sampling train consists of a personal sampling pump with an attached rotameter which has been calibrated with a representative charcoal sampling tube in the line.

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<ol style="list-style-type: none"> 2. The charcoal sampling tube (Figure 14-1) is a 7-cm. long, 6-mm. O.D., 4 mm. I.D. glass tube with both ends flame sealed. Starting from the end which is exposed to the atmosphere to be sampled, it contains a plug of silylated glass wool, 100 mg. of 20/40 mesh activated charcoal prepared from coconut shells and fired at 600°C prior to packing, a 2-mm. urethane separator, a 50 mg. backup section of activated charcoal and a 3-mm. urethane plug at the end. 3. Immediately before sampling, the ends of the tube are broken to provide an opening at least one-half the internal diameter of the tube (2 mm.). Under these conditions, the pressure drop across the tube should be less than one inch of mercury at the desired flow rate of 1 lpm. 4. The backup end of the tube is attached to the rotameter by a short length of flexible hose and the sampling tube is placed in a vertical orientation. The other end of the glass tube receives the air sample directly at a flow rate of 1 lpm. 5. If available, sample 10 liters of air from a chamber containing known concentrations of benzene, chloroform, carbon tetrachloride and trichloroethylene. Alternatively, sample the laboratory atmosphere. Record the starting and ending time of sampling and the flow rate so an accurate volume can be calculated. 6. Record the temperature and pressure of the atmosphere being sampled. 7. At the conclusion of sampling, immediately cap the ends of the glass sampling tube with plastic caps. 	

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C. Learning To Use The Gas Chromatograph 0:30

1. The instructor will demonstrate how the gas chromatograph variables are set. Typical conditions for this analysis, using a Perkin Elmer Model 900 Gas Chromatograph with a 10:1 stream splitter are:

Attenuation----- 16 x 1
Helium Carrier Gas Flow-----85 cc./min. (70 psig)
Hydrogen Gas Flow (Detector)----- 65 cc./min. (24 psig)
Air Flow (Detector)-----500 cc./min. (50 psig)
Injector Temperature -----200°C
Manifold Temperature (Detector)-----200°C
Oven Temperature (Isothermal) -----100°C

Other chromatographs may require somewhat different settings for optimum performance. The 100°C isothermal oven temperature is not necessarily optimum for each solvent but it has been shown to provide a clean separation of the four compounds to be determined in about 5-6 minutes. Lower temperature would provide greater separation but it would also require more time which is impractical in this laboratory exercise. Temperature programming is also impractical due to the time required to readjust temperatures.

2. The instructor will demonstrate the procedure for injecting a sample using a "Demonstration Standard" of the same composition as your Standard D (to be prepared in Section E). To eliminate difficulties arising from blowback or distillation within the syringe needle, the solvent flush injection technique is used. This works as follows:
 - A 10- μ l. syringe is flushed with spectroquality CS₂ several times to wet the barrel and plunger.
 - The syringe is filled with solvent to the 3.0 μ l. mark. Actually there is more than 3.0 μ l. in the syringe due to the volume of the needle.
 - After withdrawing the needle from the solvent, the plunger is drawn back to a setting of 3.2 μ l. to remove solvent from the needle.

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- . The needle is then immersed in the sample and the plunger drawn back to 8.2 μ l. This introduces 5.0 μ l. of sample with a 0.2 μ l. space between the solvent and the sample.
 - . After the needle is removed from the sample, the plunger is drawn back to 8.5 μ l. to remove sample from the needle tip where it could be lost by evaporation.
 - . The sample is injected into the chromatograph with a smooth but relatively rapid depression of the plunger to avoid spreading within the column. The solvent serves to rinse the needle and insure complete injection of the sample. This procedure should provide peak areas which are reproducible to within 3% on successive injections.
3. Each student should practice making a sample injection.
 4. Note that peak areas can be recorded directly if the instrument is equipped with an integrator. Alternatively, the areas may be estimated from the strip chart recorder tracings using a planimeter or by multiplying the height times the width at one-half peak height (assumes a triangle).

D. Preparation of Samples for Desorption Efficiency 1:00

1. Obtain from the instructor, three Parafilm sealed tubes to be used for desorption studies. Two of these, each labeled Y, contain amounts of the four organic solvents equivalent to the amounts in Standard B (See section E) adsorbed on 100 mg. of charcoal. The third tube labeled Z is the blank and contains 100 mg. of charcoal from the same lot but no added organic solvents.

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2. Obtain from the instructor a conventional sampling tube as used in the demonstration (Section B) which is labeled X. This tube contains a known amount of the four solvents. Because it was prepared in advance, there likely has been some redistribution of the adsorbed solvents, especially the more volatile ones. Consequently, it is possible that two-thirds of the adsorbed solvents will be in the front section (100 mg. of charcoal) and one-third in the backup (50 mg. of charcoal). Both the front and backup sections should be individually analyzed.
3. Obtain from the instructor about 1 ml. of Mixture S in a 2.5 ml. graduated glass stoppered microcentrifuge tube.
4. Pipet 0.50 ml. of spectroquality CS₂ into each of seven 2.5-ml. graduated microcentrifuge tubes and immediately stopper.
5. Into two of the centrifuge tubes, add the charcoal from the tubes labeled Y. Restopper and immediately note the total volume. Label the tubes Y₁ and Y₂.
6. Into another centrifuge tube, add the charcoal blank from Z. Again note the volume and label.
7. Score the sample tube X with a file in front of the first section of charcoal and break open. Remove and discard the glass wool and immediately pour the charcoal into a centrifuge tube; note the total volume; and label X_F. Remove the urethane separator and pour the backup section of charcoal into another centrifuge tube; note the total volume; and label X_B.
8. During a 30-minute desorption period, agitate the charcoal in the five tubes. If necessary, use a fine wire to push any charcoal from the walls down into the CS₂.

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<p>9. Into each of the last two centrifuge tubes, pipet 7.0 μl. of Mixture S. Ideally, you would use the same syringe as used to prepare the desorption samples Y, but this is not possible in this laboratory. Consequently, some additional error may be introduced by differences in pipets. The concentrations of the solvents in these two tubes should be the same as in Standard B (Section E). Hence, they should be labeled B' and B''.</p> <p>E. Preparation of Calibration Standards 0:30</p> <ol style="list-style-type: none"> 1. Place approximately 5 ml. of spectroquality CS₂ into each of two 10-ml. volumetric flasks. Label one <u>B</u> and the other <u>D</u>. 2. Into volumetric B, pipet 0.14 ml. of Mixture S. Keep flasks stoppered as much as possible to avoid losses by evaporation. Mix; dilute to the mark with CS₂; and mix again. The pipet may be conventional graduated glass pipet or a 500- μl. syringe. 3. Into volumetric D, pipet 0.28 ml. of Mixture S and proceed as in (2.) above. 4. Pipet 5.00 ml. of standard B into a 10-ml. volumetric flask labeled A. Dilute to the mark with CS₂ and mix. 5. Pipet 5.00 ml. of standard D into a 10-ml. volumetric flask labeled C. Dilute to the mark with CS₂ and mix. 6. The concentrations of the four organic solvents in each of the standards, expressed in units of mg./0.5 ml. CS₂, are given below. The concentrations for standard B are near the federal standards for 10 liters of air collected and desorbed into 0.5 ml. CS₂ except for carbon tetrachloride where the level was made higher than the federal standard to optimize the ease of the measurements. 	

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Concentration in mg./0.5 ml. CS₂

<u>Standard</u>	<u>Carbon Tetrachloride</u>	<u>Benzene</u>	<u>Trichloroethylene</u>	<u>Chloroform</u>
A	0.80	0.44	2.56	1.12
B	1.59	0.88	5.12	2.24
C	2.39	1.32	7.68	3.36
D	3.18	1.76	10.24	4.48

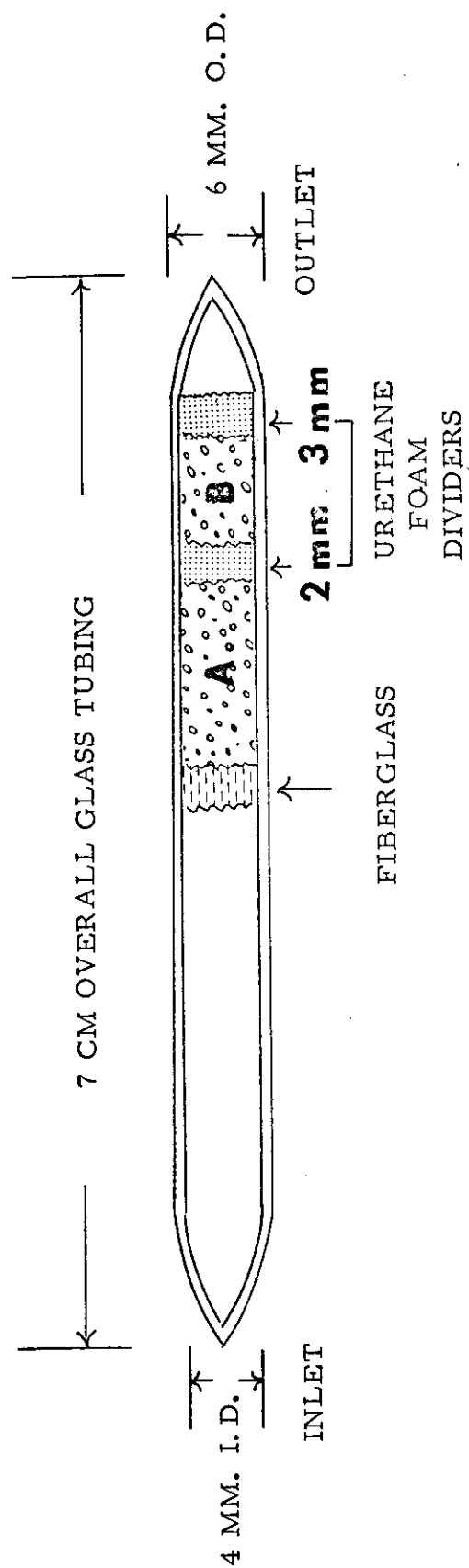
F. Analysis of Standards and Samples 1:45

1. Use the solvent flush injection technique learned in Section C to introduce a 5 μ l. portion of Standard D into the chromatograph. The components should elute in the order: carbon disulfide, carbon tetrachloride, benzene, trichloroethylene and chloroform last. Insure that each of the latter four peaks are on scale and at least one-half of full-scale for the highest standard. If any peak is less than half-scale, change the attenuation for that peak. Similarly, increase the attenuation for any peak that is off-scale.
2. Normally, at least duplicate and preferably triplicate injections would be made for each sample. Due to time limitations, only a single injection will be made for each standard and sample.
3. Prior to analysis of the samples containing charcoal in the 2.5-ml. graduated microcentrifuge tubes, readjust the total volumes to the values recorded in Section D, if necessary.
4. Analyze all standards and samples, in random order. If the instrument is equipped with a digital integrator, record areas directly in the appropriate table. If a recorder is used, measure area with a planimeter or measure height and width at half height.

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<p data-bbox="140 254 802 285">G. Calculations and Data Analysis 0:45</p> <ol style="list-style-type: none"> <li data-bbox="212 368 1275 399">1. Plot calibration curves for each of the four organic solvents. <li data-bbox="212 482 1422 586">2. Compare the areas for B' and B'' with standard B which was prepared in large volume. Use the results for B' and B'' to obtain averages for the B standard. <li data-bbox="212 669 1444 814">3. Determine the desorption efficiency by comparing the averages for the B standards to the averages for the blank corrected Y samples according to the equation given on the form. This determination must be made for each organic solvent on each new batch of charcoal. <li data-bbox="212 897 1472 1073">4. Read the weights in mg. for the unknowns X_F and X_B from the calibration curves. Correct the mg. in X_F by subtracting Blank Z and X_B by subtracting one-half of Blank Z (50 mg. charcoal instead of 100 mg.). Add blank corrected mg. in X_F to blank corrected mg. in X_B and divide the sum by the desorption efficiency. <li data-bbox="212 1156 1444 1218">5. Compare the results to the known values supplied by the instructor and to the values obtained by the rest of the class. <li data-bbox="212 1301 1325 1363">6. Express the concentrations in the unknown in ppm. using the V_s value obtained in Section B. 	

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A - 100 MG. 20/40 MESH ACTIVATED CHARCOAL

B - 50 MG. 20/40 MESH ACTIVATED CHARCOAL

Figure 14 - 1. Charcoal Tube

Demonstration of a Sampling Train for Organic Solvents
(Section B)

Name: _____

Date: _____

Time Start: _____

Time End: _____

Collection Time: _____ minutes

Flow Rate: _____ liters /minute

Total Volume Uncorrected (V) : _____ liters

Temperature (T): _____ °C

Pressure (P): _____ mm Hg

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

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

$$V_s = \underline{\hspace{2cm}} \times \frac{\underline{\hspace{2cm}}}{760} \times \frac{298}{\underline{\hspace{2cm}} + 273} = \underline{\hspace{2cm}} \text{ liters}$$

Analysis of Standards and Samples (Section F)

Std. or Sample	Carbon Tet. Peak Ht. (mm.)	Peak Width Half Ht. (mm.)	Carbon Tet. Area	Benzene Peak Ht. (mm.)	Peak Width Half Ht. (mm.)	Benzene Area
Std. A						
Std. B						
Std. C						
Std. D						
Spl. Y ₁						
Spl. Y ₂						
Avg. Y						
Blank Z						
Std. B'						
Std. B''						
Avg. B						
Spl. X _f						
Spl. X _b						
	Trichloroethylene		Trichloroeth- ylene	Chloroform		Chloroform
Std. A						
Std. B						
Std. C						
Std. D						
Spl. Y ₁						
Spl. Y ₂						
Avg. Y						
Blank Z						
Std. B'						
Std. B''						
Avg. B						
Spl. X _f						
Spl. X _b						

Preparation of Samples for Desorption Efficiency
Determination and Preparation of Unknown
(Section D)

Label on Centrifuge Tube

Total Volume Charcoal Plus CS₂

Y₁

_____ ml.

Y_z

_____ ml.

Z

_____ ml.

X_f

_____ ml.

X_b

_____ ml.

Calculations and Data Analysis
(Section G)

1. Desorption efficiency = $\frac{\text{Average Sample Area (Y)} - \text{Blank Area (Z)}}{\text{Average Standard Area B}}$

	<u>Carbon Tet.</u>	<u>Benzene</u>	<u>Trichloroethylene</u>	<u>Chloroform</u>
Avg. mg. Y	_____	_____	_____	_____
mg. Z	_____	_____	_____	_____
Y - Z (mg.)	_____	_____	_____	_____
Avg. mg. B	_____	_____	_____	_____
$\frac{\text{Avg. Y-Z}}{\text{Avg. B}}$	_____	_____	_____	_____

This last value is the desorption efficiency (DE) for each.

2. Calculation of weights in the unknown.

mg. X_f (curve)	_____	_____	_____	_____
mg. Z (curve)	_____	_____	_____	_____
$X_f - Z = X_{fc}$ (mg.)	_____	_____	_____	_____
mg. X_b	_____	_____	_____	_____
mg. $Z/2$	_____	_____	_____	_____
$X_b - Z/2 = X_{bc}$ (mg.)	_____	_____	_____	_____
$X_{fc} + X_{bc} = X_{tot}$	_____	_____	_____	_____
$\frac{X_{tot} \text{ (mg.)}}{\text{DE}}$	_____	_____	_____	_____

This last row of values represent the fully corrected weights (mg.) of each solvent in the unknown.

3. Conversion of Values to ppm. based on Vs from Section B.

ppm	=	$\frac{\text{mg. of compound}}{\text{Vs (liters)}}$	x	$\frac{24,450}{\text{molecular wt.}}$	
ppm Carbon Tet.	=	_____	x	$\frac{24,450}{154}$	= _____
ppm Benzene	=	_____	x	$\frac{24,450}{78.1}$	= _____
ppm Trichloro-ethylene	=	_____	x	$\frac{24,450}{131.4}$	= _____
ppm Chloroform	=	_____	x	$\frac{24,450}{119.4}$	= _____