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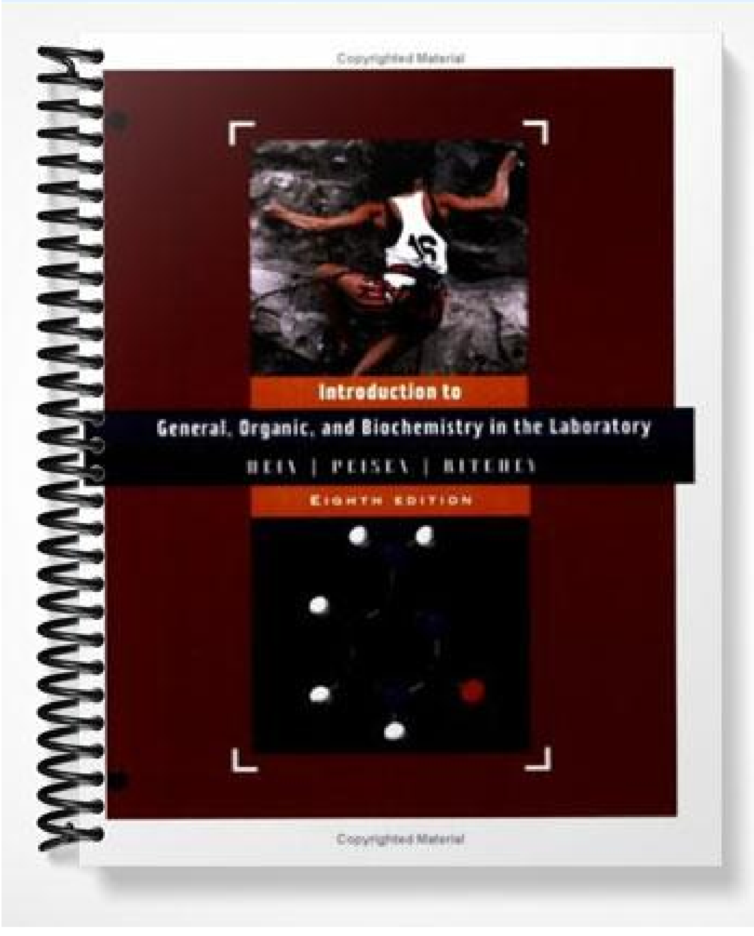

Instructor's Manual for

Introduction to General, Organic, and Biochemistry in the Laboratory

HEIN | PEISEN

EIGHTH EDITION

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INSTRUCTOR'S MANUAL FOR

INTRODUCTION TO GENERAL, ORGANIC, AND BIOCHEMISTRY IN THE LABORATORY

EIGHTH EDITION

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NOTE: For your ease of use, the KEYS in Section E match the page numbering in the student lab manual (even though this does not match the actual page numbers of this manual). Therefore, the KEY for the Report for Experiment 1 (Section E) is on pages 9 and 10, and the KEY for the Report for Experiment 2, which follows the Report for Experiment 1 directly, starts on page 19, etc.

A. LABORATORY MANAGEMENT

This section is not intended to tell you how to manage your laboratory program. However, we have learned a lot over many years of working with beginning chemistry students and some practices work better than others. Some of these practices are included here.

1. At the beginning of each term,
   a. We give each student a copy of the schedule of experiments. Wherever possible we prefer to schedule experiments which support the ongoing lecture content. Copies of this schedule are also posted on the laboratory bulletin boards.
   b. A system for replenishing supplies as they run out should be in place to avoid last-minute problems with availability.
2. All sections of a given course do the same experiment during the same week insofar as possible. During weeks with holidays, this is accomplished by omitting an experiment for some sections or assigning a "Review Session" or "Recitation" for others.
3. We begin each session with a prelab discussion period. During this time we provide an overview of the experimental objectives, discuss principles, demonstrate techniques, emphasize safety and waste disposal precautions, and answer student questions pertinent to the current experiment. The length of these discussion periods averages about 25 minutes.
4. In order to make laboratory work more than an exercise in manipulation, we grade all report forms and schedule at least two lab exams per semester, a midterm and a final exam.
5. Report forms are collected for every experiment on the same day the experiment is performed. This encourages students to prepare before coming to each session, to budget their working time more effectively, and to focus on the underlying principles of the experiment as they answer questions and do the problems on the report form. Exceptions to this policy are made at the discretion of the instructor when the discussion, experimental work, or examinations consume more than the allotted time.
6. All report forms are graded using student readers when available. The answer keys (Section E) are setup to match with the student report forms and facilitate rapid evaluation. With this edition, we have provided sample data for all experiments. We require students to maintain a file of and use their graded report forms to study. Students are required to hand in these files at the end of the term (or when dropping the class).
7. We emphasize the proper disposal of wastes constantly and consistently right from the start. The procedures provide specific instructions every time a student is faced with a disposal decision.

   WASTE DISPOSE OF PROPERLY

   These instructions are emphasized by the waste icon (left margin). The waste collection containers we use for each experiment are listed in this Instructor's Manual in Section C (Notes on Individual Experiments at the end of each discussion) and in the lab manual, Appendix 8. Waste disposal is an important component of the Chemical Management Programs for our institutions, and is a line item in the department budgets. In the final analysis, each chemistry department will and should have their own regulations for disposal of wastes.

B. EVALUATION OF EXPERIMENTS

1. We try to grade and return report forms the week after each experiment is done. Each student response on the report form is assigned a point value: qualitative observations, properly recorded quantitative data, calculation setups and problem solutions, are all included. Unless this is done, some students begin to neglect the descriptive parts of the

experiment, get careless with significant figures in measurements, and neglect the calculation setups. When both descriptive and quantitative parts are included on a report form, the quantitative parts are generally weighted more heavily. Lab partners are encouraged to work together but to put all answers into their own words.

2. Answer Keys for all the experiments are given in Section E. We have included *sample* data for many experiments, thus student data will not match the key exactly. When student graders are used, we find they need instruction and/or supervision occasionally concerning their judgment of student answers.
3. It is not our intent to assign point values to parts of experiments. This evaluation is the prerogative of each instructor and will vary due to individual preferences and emphasis. Where our experience has contributed to concrete evaluation, we have given a grading scale and suggested limits for certain data. These suggested evaluations are included in Section C, Notes on Individual Experiments.

C. NOTES ON INDIVIDUAL EXPERIMENTS AND WASTE REQUIREMENTS

FIRST LABORATORY CLASS MEETING

At the first laboratory session we assign lockers and supervise the checking out of equipment by students. We discuss every item with its rationale in the Laboratory Rules and Safety Procedures in the lab manual emphasizing that no student can participate without eye protection. We point out special equipment and features of the laboratory and demonstrate special items of safety equipment. We go over waste disposal procedures and familiarize students with the location of waste containers. For the protection of the instructor, the school, and the student, it is becoming common practice for each student to sign a safety contract stating their knowledge and understanding of these rules. Thus, when this session is complete, every student signs our contract which is filed in the department office. A sample of this contract is provided below. If you choose to require a safety contract for your students, this sample can be copied or modified to suit your needs. If time permits during this first class meeting, Part of Experiment 1 is begun.

> I have read and understand the Laboratory Rules and Satefy Procedures in my laboratory manual. I accept the responsibility for following all of these rules and procedures in order to maintain a safe laboratory environment for myself and others.
>
> Course ____________________ Instructor ____
>
> Student Signature __________________________________ Date ____________________
>
> Do you wear contact lenses? yes ________ no ________

EXPERIMENT 1: Laboratory Techniques

Since most of the students have had little or no prior laboratory experience, it is desirable to demonstrate the following techniques:

1. Adjusting the burner.
2. Cutting, fire-polishing, and bending glass tubing (specimens of properly fire-polished and bent tubing are made available for student inspection).

3. Inserting glass tubing into and removal from rubber stoppers. Care is taken to point out the safety advantages of (a) fire polishing, (b) proper use of a suitable lubricant (glycerol), and (c) the technique of gripping the tubing close to the stopper. NOTE: A No. 3 cork borer is a useful tool for removing 6 mm glass tubing from stoppers.
4. Folding filter paper to form a cone and seating the cone in a funnel.
5. Using a stirring rod in transferring a liquid (decanting from a beaker to a filter).
6. We like to have students see the actual MSDS sheets for the chemicals used in this experiment so they realize that the waste regulations have a valid rationale. This is not a formal part of the experiment, but the sheets are made available.

Note that the report calls for the instructor to evaluate student-prepared glassware.

Waste requirements:
Two waste containers are needed: (1) a bottle for Waste Heavy Metal, Pb^{2+}, and (2) a jar for filter paper with PbI_2.

EXPERIMENT 2: Measurements

We consider this a vital experiment for getting students off to a good start in several areas:

1. basic measurements and their relationship to significant figures
2. problem setups and dimensional analysis
3. the use of rules for significant figures in calculations
4. the difference between precision and accuracy in measurements, and
5. following directions

Each instructor will probably want to emphasize certain of these areas in the prelab discussion and report grading. Review of this experiment when the report is returned is particularly valuable. We refer students to Study Aids 1 and 5, and assign Exercises 1 and 2 in connection with this experiment. For help with the use of a scientific calculator, we refer them to Study Aid 4.

Since various types of balances are used in different schools, we have omitted detailed instructions on the use of the balance. The instructor should demonstrate weighing procedures. Any tips that will speed up the students' weighings will be most valuable in this and in many subsequent experiments. For economy of weighing time it is especially important to point out the difference between weighing an exact quantity (utmost precision) and weighing an approximate quantity.

The directions for the Eighth Edition are written for an electronic balance with the uncertain digit in the 0.001 place. We also introduce the "tare" function and we require that all measurements be recorded to include one uncertain digit. Rounding is done only when a measurement is used in a calculation and the rules of significant figures are applied. The degree of precision in the answer keys must be adjusted for the equipment used by your students.

When the number of balances is limited, students are apt to be waiting in line to use the balance for Part B. We suggest that some students begin with Part B and others delay Part B until the balances are more available.

Unknowns:
We use metal slugs, 7/16 to 1/2 inch diameter by approximately $1\frac{3}{4}$ inches long. Slugs should be somewhat variable in length and should be numbered.

Materials for slugs: Aluminum, brass, and steel (other materials can be used).

Evaluation:
For this experiment, in particular, evaluation is made on (1) precision of data, (2) correct problem setups, (3) density determinations, and (4) units in Part E. No precision is checked for Parts C and D.

Permissible density ranges (suggested).

1. Water	0.94	–	1.06	g/mL
2. Rubber stopper	1.1	–	1.5	g/mL
3. Unknowns				
(a) Aluminum	2.2	–	3.2	g/mL
(b) Brass	8.3	–	9.3	g/mL
(c) Steel	7.3	–	8.3	g/mL

Waste requirements: none

EXPERIMENT 3: Preparation and Properties of Oxygen

Since this is the first experiment involving the collection of a gas by water displacement, the use of a pneumatic trough, including the handling of inverted bottles of water, should be demonstrated.

This is also the first experiment in which the students are asked to write chemical equations. It therefore, affords an opportunity to explain and discuss the concept of chemical equations during the introductory discussion period. Refer students to Study Aid 2.

Safety Precautions:

1. Hydrogen Peroxide:
   a. Nine percent hydrogen peroxide may be prepared from commercially available 30 percent H_2O_2. (Purchase the peroxide with added preservative.)
   b. Use gloves and eye protection when handling 30 percent H_2O_2, since it is a hazardous chemical.
   c. Students should not be allowed to handle 30 percent H_2O_2, but may safely handle the 9 percent solution. However, any skin area coming in contact with the 9 percent solution should be washed with water immediately.
2. Students sometimes have trouble getting the steel wool to burn in oxygen. Emphasize that the steel wool must be quickly transferred from the flame to the bottle of oxygen and, in fact, must be glowing when it is inserted.
3. Students should be reminded not to look directly at the flame produced by burning magnesium.
4. The Büchner funnel-vacuum flask apparatus for the separation of the MnO_2 in the generator flask from the H_2O_2 mixture should be demonstrated before students begin the experiment. One of these setups at a sink that is accessible to everyone in the class is probably adequate for 24 students who work in pairs. The filter should be monitored and changed as needed when it is being used by many students. The level of the reacted H_2O_2 mixture in the vacuum flask should also be monitored and emptied into the sink as needed.
5. Instructor Demonstrations: The spontaneous combustion that occurs when water is added to sodium peroxide on cotton makes a spectacular demonstration but should be conducted with caution. Test the sodium peroxide in advance to make sure it is still active.

Stability of Hydrogen Peroxide Solution

Thirty percent H_2O_2 should be stored in the refrigerator. Nine percent H_2O_2 is stable for long periods of time when stored in the refrigerator but decomposes at room temperature. Store the 9 percent solution in brown bottles. If the 9 percent solution has been standing at room temperature for several days it may be necessary to use more than 50 ml to obtain the required amount of oxygen. It is advisable to prepare the 9 percent solution shortly before use.

Waste requirements:
A bottle for recycling unreacted 9 percent H_2O_2 should be provided, the Büchner funnel-vacuum flask should be setup for disposal of MnO_2.

EXPERIMENT 4: Preparation and Properties of Hydrogen

In addition to illustrating some of the properties of hydrogen gas, this experiment provides qualitative experience with pH and the reactivities and reaction rates of different acids with metals. Additional experience in handling simple chemical equations is also provided.

If Experiment 3 has not been done, refer the students to it for directions on using a pneumatic trough and gas collecting bottles. Also refer the students to Study Aid 2.

A great deal of student time can be saved by preparing a sample bottle containing about 10 grams of mossy zinc to help students approximate this amount. Since the unused zinc is collected for reuse, it doesn't matter if more than 10 grams is used.

It is important to emphasize the distinction between combustible substances and substances which support combustion.

Because of the great tendency of hydrogen gas to escape, success with the tests of Part D is dependent on lifting the bottle straight up from the table top **without** tilting it to one side.

Safety Precautions:

1. It is advisable for the instructor to dispense one cube of sodium metal to each student. These cubes should be no larger than 4 mm on an edge.
2. Remind students not to look directly into the test tube when sodium and water are reacting. The escaping hydrogen may ignite and splatter unreacted sodium metal and sodium hydroxide solution.
3. Remind the students to keep lighted burners and all other flames away from the hydrogen generator.

Waste requirements:
(1) A container for unreacted metal strips is needed, and (2) a recycling jar for used zinc (rinsed with water) should be provided.

FIRST GRAPHING SESSION

Early in the semester before graphing skills are required for experimental data, we devote a full laboratory session to graphing, both by hand and by computer. Since Experiment 5, Calorimetry and Specific Heat involves a graph, the graphing session is usually done before Experiment 5. You may choose to do this in a different sequence or have students learn this on their own. During this session Study Aid 3 (SA3) in its entirety is completed:

1. Plotting two variables by using graph paper; two practice graphs are included in SA3.
2. Completion of Exercise 9 on plotting data and reading graphs.
3. Completion of a computer graph of data in SA3 using Excel, Chart Option (within Microsoft Office 2000 or XP.) If your facilities lack computers in the laboratory, students can complete this component in a computer lab or at home; the Chart Option can also be accessed through MS Word. The directions can be applied to both Windows-based PC computers and MacIntosh. System requirements are described in the Study Aid.

At first the computer graphing requirements may be difficult for some students because they have little or no experience with computers. These students need some help and support as they learn how to manipulate a mouse and gain confidence with the keyboard. They benefit from working with more sophisticated students and are usually transformed by successful completion of their first graph.

EXPERIMENT 5: Calorimetry and Specific Heat

This experiment provides support and reinforcement to several important areas:

1. determination of specific heat of a known substance by using a calorimeter;
2. completion of two trials of a procedure to determine the precision of the specific heat measurements;
3. comparison of the experimental specific heat to a theoretical value to determine the accuracy of the specific heat measurements;
4. practice of graphing skills using atomic mass vs. specific heat data provided in the discussion.

We use the same metal slugs that were used for the density measurements in Experiment 2. Sometimes (on a very dry day) there is enough static electricity generated by the styrofoam cups that the electronic balances are erratic. It is good to have a low-tech balance available. Common sources of error include:

1. test tube in the boiling water should not rest on the bottom of the beaker.
2. boiling water should not evaporate below the top of the metal slug in the test tube.
3. temperature of the thermometer in the boiler should not be greater than 100°C.
4. transfer of hot metal to the calorimeter must be done quickly as heat is lost to the surroundings very quickly. After the transfer of the metal into the calorimeter, the water should be stirred while monitoring the temperature.
5. the volume of water in the calorimeter should be just enough to cover the metal sample. Too much water results in a very small temperature change because of the high water mass absorbing the heat.

For Trial 2, the water should be colder than Trial 1 to illustrate that the temperature change experienced by the water in the calorimeter, not the initial temperature, is the essential quantity in determining the heat absorbed by the water. A one-liter bottle of distilled water in an ice bath will provide enough water at 5–10°C for an entire class.

The discussion provides a sample calculation as a template for the experimental calculations and we insist on students showing setups on the report form as well as answers.

Evaluation:
High accuracy (i.e., agreement with the theoretical value for the metal sample) is not a priority in the experiment. We look for an improvement in accuracy from the first trial to the second trial. Graphing skills, problem setups, and insight into heat concepts are the priorities here.

Waste requirements: none

EXPERIMENT 6: Freezing Points—Graphing of Data

This experiment has several objectives:

1. To provide practice in taking and plotting data using graphing skills learned in Study Aid 3. Graphs are done by hand or by computer.