

Organic chemistry lab report assignment



CHEM 2123 and 2125 – Organic Chemistry Lab I and II GUIDELINES FOR

WRITING LAB REPORTS INTRODUCTION Writing reports in organic chemistry lab may differ from the way it's done in general chemistry. One goal of this course is to introduce you to the record keeping methods used in research labs. Such methods are designed to organize experimental data in a format similar to that required for publication in major scientific journals. Here are some important considerations that apply in research settings. 1. Your work is unique, meaning that you might be the only person performing certain experiments. 2. Research is an ongoing process.

The projects assigned to you will likely continue after you leave. People assigned to those projects will need to access your records for reference, or to attempt to reproduce work you did. Permanence is a characteristic of properly kept research records. 3. It is therefore important to follow guidelines for clear writing. In addition to writing legibly, you should use permanent ink. A ballpoint pen is adequate for this purpose, but a pencil is obviously not. 4. Write your records by hand, either while conducting the experiment, or immediately afterwards. This is important to avoid forgetting important details.

You will make mistakes, but that is less important than recording data while it's still fresh in your mind. 5. Mistakes are not to be erased. A "mistake" might turn out to be important information later. You and others must be able to read what you wrote. If you make a mistake, draw a line across the text, but make sure it remains legible, like this. 6. Remember: your lab notebook is the first line of documentation that exists of your experiments. It is important that this information be as fresh and accurate as possible. Don't

worry if it's imperfect. You will have occasion to produce a formal, well written report later.

It may be a thesis or a scientific publication, but it will likely bring together several experiments with a common theme. Your lab notebook will be the primary source of data for such publication. 7. Do not write on loose sheets of paper, or even in spiral notebooks from where pages can be torn. Use a hardbound notebook, with numbered pages, that makes carbon copies as you write. That makes it easy to spot missing pages, and it gives you two sets of records that you can keep in separate locations in case one of them gets lost or damaged. 8. Do not tear pages from a research notebook unless they're damaged or threaten the integrity of the notebook (by catching acid, for example). If both original and copy get damaged, try to reproduce their contents the best you can, with a note explaining the absence of the originals. TYPES OF REPORTS HANDLED IN THESE COURSES In the organic chemistry lab course there are two types of reports, which are all graded on a 100 point scale. One type comprises what's commonly known as "lab reports," that is, records referring to experiments performed in the lab. The other type refers to special assignments that are completed outside the laboratory.

The format and contents of special assignments depends on their nature. They are specified in the syllabus, or at the time the assignment is made. Examples are the MSDS assignment in organic lab I, or the Chemical Literature assignments in organic lab II. Lab reports, on the other hand, have a set format. They can be of two kinds, depending on whether they refer to experiments involving physical operations or chemical preparations

(synthesis). 2 LAB REPORTS FOR PHYSICAL OPERATIONS, OR TECHNIQUE EXPERIMENTS This type of report refers to lab work whose main objective is to learn, demonstrate, or perform a physical operation.

Physical operations do not change the chemical nature of the substances involved. They are typically conducted as part of a synthesis and are frequently of two types: 1. Purification techniques, or separations. These operations are designed to isolate a pure substance from a mixture. Examples are crystallization, extraction, and distillation. 2. Characterization techniques. These operations are designed to return information that can be used to identify a substance. Examples are the determination of physical constants such as melting point, boiling point, or density, and instrumental techniques such as infrared (IR) spectroscopy.

Sometimes these techniques are referred to as analytical techniques. LAB REPORTS FOR CHEMICAL PREPARATIONS, OR SYNTHESIS This type of report refers to experiments whose main goal is to prepare a pure substance from specific starting materials. This necessarily involves a chemical transformation, or reaction. In the simplest case, there is only one step. The starting materials are combined and a product forms. This product is isolated, purified, and characterized, producing the final outcome of the experiment.

In a multistep synthesis, the product of the first step is used as a starting material in a second step, and so on, until a final product is obtained. No multistep syntheses are performed in organic lab I. Some two step syntheses are performed in organic lab II. FORMAT AND GRADING CRITERIA FOR

PRELABS AND LAB REPORTS The general format for most prelabs and lab reports is given below. Specific differences with examples will be discussed later. You are expected to include the following items in your lab notebook. All prelabs and lab reports are graded on a 100 point scale with the following distribution.

INDIVIDUAL PRELAB (100 pts) Due at the beginning of each experiment. 1

NAME, EXPERIMENT #, TITLE, DATE, INSTRUCTOR, and SECTION

INTRODUCTION a. Objectives of the experiment b. Table of physical constants c. Chemical equations and reaction mechanisms (for synthesis only) d. Diagram or sketch of setup used (if it serves for illustration purposes or to enhance clarity) **EXPERIMENT OUTLINE** (abbreviated list of experimental steps) or **FLOW CHART** (diagram clearly showing the flow of operations) 60 points Equally distributed among the items actually required for specific experiments. 0 points Include either outline or flow chart, not both. 2 3

GROUP LAB REPORT (100 pts) Due a week after concluding the experiment.

1 2 3 4 5 NAMES OF GROUP MEMBERS, EXPERIMENT #, TITLE, DATE, INSTRUCTOR, and SECTION **OBSERVATIONS OF PHYSICAL CHANGES** **TABLE OF RESULTS** (with supporting materials) **RESULTS, DISCUSSION, and CONCLUSIONS** **ASSIGNED PROBLEMS** (from the textbook) 30 30 30 10 points

" " " 3 **INTRODUCTION** – Depending on the type of experiment being performed, this section may contain the items shown below. The first two are relevant to all experiments.

The last two are relevant to some experiments. If you cannot find some of the required information in textbooks and common sources such as MSDS or the Aldrich catalog, simply indicate so. If the information is not pertinent,

write N/A for not applicable. If it is unknown, indicate so. a. b. c. d. Objectives of the experiment Table of physical constants Relevant chemical equations and reaction mechanisms (in the case of synthesis) Diagram or sketch of setup used (if it serves for illustration purposes or to enhance clarity) Objectives can be learning objectives and/or experimental objectives.

For example, in a crystallization experiment the learning objective is to illustrate the technique of crystallization by having the student perform the steps involved. But the experiment objective is to purify a substance by crystallization and prove that it is reasonably pure by its melting point. A table of physical constants should include the following for every major chemical used in the experiment: chemical structure, molecular weight, boiling and/or melting point, density, and safety hazards such as flammability and toxicity.

Under safety hazards, indicate the source of information with a footnote (Aldrich catalog, MSDS, etc.). If the substance poses special hazards, make a note under the table regarding safe handling and emergency procedures in case of contact. An MSDS must always be consulted before handling hazardous substances. Synthesis reports must include chemical equations. Include molecular mass and amounts used, in grams and in moles, for each reactant. This will enable you to find out which is the limiting reagent when the time comes to calculate the percent yield of the product.

If the mechanism of the reaction is known (given in your book or in class), make sure to include it too. A picture can save you many words. If it serves an illustrative purpose, the introduction can also include a diagram or sketch

of the setup used, such as a distillation apparatus. It doesn't have to be a work of art and it doesn't need to be done every time a distillation is performed. If you do distillations again, simply refer the reader to the first experiment where the setup was used, or to a source such as an article or book. Examples of different parts of a lab report are given at the end of this guide and in your textbook.

EXPERIMENT OUTLINE or FLOW CHART OF OPERATIONS – An experiment outline is a list of the steps that go into performing an experiment, written in abbreviated form. The main purpose of this list is to save time by serving as a quick reference while performing the experiment. Most experimental descriptions in the textbook tend to be verbose and difficult to follow, usually covering several pages. By distilling these descriptions into a list of steps the student is able to perform the experiment faster, requiring less assistance from the instructor(s).

A flow chart of operations (like those used in engineering) is similar to a list of steps, except that it relies on pictures, arrows, and brief descriptions rather than words. The choice of outline or flow chart depends strictly on the student's personal preference, although some experiment may lend themselves better to one or the other.

OBSERVATIONS OF PHYSICAL CHANGES – As you perform the experiment, some physical changes may or may not happen. For example, when you combine two substances, a change in color might occur, the solution might get hot, a precipitate may form, or bubbles might appear.

The rule of thumb is to record any changes that can be detected with the senses. While most of them are visual (e. g. a change in color or formation of a precipitate), some are tactile (a change in temperature), or even auditory (an explosion, which hopefully will not happen in this course!). On occasion a smell might be detected, and this qualifies as a physical observation. Keep in mind that in this section all you do is record such observations as objectively as possible, but you do not try to infer a meaning from them. This is done in the results and conclusions section, described ahead.

TABLE OF RESULTS AND SUPPORTING MATERIALS – This is similar to the table of starting materials but it shows data that pertain to the products or results of the experiment. Supporting materials is any physical evidence such as spectral charts, chromatograms, or instrument readouts. Sometimes there are no supporting 4 materials. For example when you take a melting point you simply read it from a temperature scale, but the instrument doesn't produce any chart or physical record of it. On the other hand, when you run a chromatographic plate, you have the plate as evidence of the results.

A report referring to a physical operation should contain the parameters indicative of the success of the operation. For example, in a purification procedure, give the parameters that indicate the degree of purity of the substance (e. g. melting point, IR spectrum). Also give the percent recovery of pure substance relative to impure substance, complete with relevant calculations. A report referring to a reaction or synthesis should contain any parameters that indicate you obtained the expected product. Examples are melting point or spectral data.

Include the percent yield with relevant calculations (make sure that you know how to identify the limiting reagent when one of the reactants is in excess). **RESULTS, DISCUSSION, AND CONCLUSIONS** – This is where subjective interpretation comes in. By now you have gathered a certain amount of data, figures, charts, observations, etc. It is time to interpret what that information means. For example, is the melting point obtained close to the expected value? If not, what could possibly be the reason? What information can be gathered from a chromatogram or a spectral chart? Does it support the results you expect to obtain?

If a precipitate forms or a change in color occurs, what can that possibly tell us? Speculations are OK as long as they can be backed up with logical arguments. Understandably, students find this the hardest part of the report to write. If you experience this type of difficulty, it might help you to go back and retrace all the parts of the lab report. Start with the introduction. Were the objectives and goals of the experiment met? If not, why not? This is where you start thinking about possible sources of error. Types of error can be systematic or random. Systematic error is a consistent error that can be detected and corrected.

This occurs most commonly when using precision equipment that requires calibration, such as thermometers, burets, and pH meters. Miscalibration can give rise to systematic error. Random error results from limitations in our ability to make physical measurements. For example, when reading a scale, different people might report slightly different results due to their subjective interpretation of the values they read between marks. Random error can also result from variations in an instrument's performance inherent in its physical

makeup. For example, electrical noise might produce slightly different readouts at different times.

Random error cannot be eliminated, but it can be reduced by improving the experiment, or by using better equipment. Next, you might want to go to section 3 and see if you can provide explanations for at least some of the physical changes observed here. For example, in a Grignard reaction, a change in color is observed as the reaction proceeds. This might be explained by invoking formation of an intermediate in the reaction mechanism. When a step calls for neutralization of excess acid with sodium bicarbonate, formation of bubbles is observed. This can be explained if you know that this reaction produces carbon dioxide gas.

As you can see from these examples, many observations can be explained if you have a good understanding of the theory of the experiment. The last section to draw from is section 4, where you have presented all the raw data that you gathered during the experiment. The term raw data refers to information that has not yet been organized or interpreted. You can start by answering the question, were the outcomes of the experiment as expected? If yes, what evidence supports this conclusion? For example, was the melting point within the range reported in the literature? If not, what does the evidence indicate that might have happened instead?

Maybe the spectrum of what you think is a product really corresponds to the spectrum of the starting materials, indicating that there was no reaction. Maybe it is a mixture of products and starting materials, indicating partial reaction. Maybe it is the spectrum of a byproduct, indicating that the

conditions of the experiment favored formation of a product other than the expected one. If the experiment worked as expected, there is little need for superfluous elaboration in this section. Remember that the quality of a report does not correlate with the amount of writing done.

It correlates with the substance and the clarity of the material presented. 5

EXAMPLES OF PORTIONS OF LAB REPORTS EXAMPLE 1: INTRODUCTION TO A

REPORT OF A PHYSICAL OPERATION In this example, the students will perform a physical operation known as crystallization. This operation is intended to purify solids containing small amounts of impurities.

INTRODUCTION. In this experiment acetanilide will be purified by crystallization from water. The learning objectives are to learn the applications of this technique and its execution, and to learn how to take a melting point. The expected outcome is to obtain a pure substance from an initially impure sample and to demonstrate its purity by recording its melting point. TABLE OF PHYSICAL CONSTANTS Substance Structure O H N CH 3 M. W. m. p. (oC) b. p. (oC) Density Safety hazards* Acetanilide 135. 17 117-115 — — Toxic, Irritant Water H2 O 18. 02 0 100 1. 00 nontoxic * Source: Aldrich catalog, 2003-04. No highly toxic substances are handled in this experiment.

In case of skin contact with acetanilide, wash affected area with plenty of soap and water. EXAMPLE 2: INTRODUCTION TO A REPORT OF A CHEMICAL REACTION, OR SYNTHESIS In this example, the students will prepare 1-bromobutane from 1-butanol in an acid catalyzed reaction.

INTRODUCTION. In this experiment 1-bromobutane will be prepared from 1-butanol in an acid-catalyzed S_N2 reaction. The main learning objective is to illustrate a synthetic application of the S_N2 reaction. The expected outcome

is to obtain a reasonably good yield of product with minimal formation of side products such as alkenes arising from elimination reactions.

CHEMICAL EQUATIONS

$$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{NaBr} \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{NaHSO}_4 + \text{H}_2\text{O}$$

M. W. amounts used

mmoles used 74.12 (185 mg) 2.50 NaBr 102.9 (300 mg) 2.92 + H₂SO₄ 98.08 (250 mg) 2.56

4.69 CH₃CH₂CH₂CH₂Br + 1-bromobutane 137.03 (342 mg) ((theoretical) 2.5 (theoretical) NaHSO₄ + H₂O sodium bisulfate 6

CALCULATIONS Calculating mass of sulfuric acid from its measured volume Density = mass (g) / volume (mL) mass = density X volume = 1.84 g/mL X .25 mL = 0.46 g

Calculating mmoles from the mass of the substances Moles = mass (g)/M. W. (g/mol) Moles of 1-butanol = .185 g /74.12 = .0025 moles = 2.5 mmoles Moles of NaBr = .300 g /102.9 = .00292 g = 2.92 mmoles Moles of H₂SO₄ = .46 g /98.08 = .00469 moles = 4.9 mmoles

Calculating theoretical yield of product The limiting reagent in this reaction is 1-butanol. The balanced equation shows that one mol of 1-bromobutane is obtained for every mol of 1-butanol used. Since we are starting with 2.5 mmoles of 1-butanol, then the theoretical yield of 1-bromobutane is also 2.5 mmoles, or .342 g. You must note the following: (a) When handling very small amounts of substances it is more convenient to use millimoles (mmoles) instead of moles. (b) We are mainly concerned with reporting the properties of organic products such as 1-bromobutane, and not inorganic products such as sodium bisulfate.

Inorganic products are typically separated and discarded. (c) Not all reagents are present in equimolar amounts. In such reactions, the limiting reagent is the one present in the smallest amounts, such as 1-butanol in this example.

TABLE OF PHYSICAL CONSTANTS Substance 1-Butanol Sodium bromide

Sulfuric Acid 1-Bromobutane Structure $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ NaBr H_2SO_4 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br}$ M. W. 74. 12 102. 9 98. 08 137. 03 m. p. (°C) -89. 8 —112

b. p. (°C) 117. 5 –100 – 104 Density (g/mL) . 81 -1. 84 1. 276 Safety hazards*

Flammable Irritant Corrosive Flammable * Sources: Aldrich catalog, 2003-04, MSDS.

All substances must be handled in the hood, wearing gloves and eye protection. Avoid contact with eyes and inhalation. Skin contact procedures: Wash affected area with plenty of soap and water. If a skin rash or a burning sensation results, see a physician immediately. Eye contact with sulfuric acid requires immediate attention. Wash with plenty of water and see a physician as soon as possible.

REACTION MECHANISM The reaction between sodium bromide and sulfuric acid generates HBr . There is a high concentration of bromide ions in this medium. The first step is a proton transfer to produce the protonated alcohol with a good leaving group (water). In the second step, bromide ion acts as a nucleophile to displace the water and form the substitution product.

(1) $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{H}_2\text{SO}_4 \rightarrow \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}_2^+ + \text{HSO}_4^-$

(2) $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}_2^+ + \text{Br}^- \rightarrow \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{H}_2\text{O}$

APPARATUS USED A common setup such as a reflux or a distillation apparatus can be simply described with words, or a reference to another source can be given. More unique setups should be drawn to the best of the student's ability.

Here is an example: A reflux setup such as the one shown on p. 198 of the Pavia textbook (4th ed.) was used. The only modification was the omission

of the gas trap at the top, since this reaction was conducted in the hood.

EXAMPLE 3: EXPERIMENT OUTLINE and OBSERVATIONS In this example, acetylglycine is being prepared by reaction of glycine with acetic anhydride.

Here is what the experiment outline and corresponding observations might look like. EXPERIMENT OUTLINE 1. 2. 3. 4. 5. 6. 1 Place 0. 10 g glycine in a 5 mL conical vial Add 0. 4 mL water and warm slightly to dissolve Add 0. mL acetic anhydride and stir mixture for about 5 min. Cool mixture in ice bath for about 10 min. Collect precipitate by suction filtration and wash with a few mL of water Spread crystals over a piece of filter paper or watch glass and air dry for 10 min. 7. Record mass and melting point of product

OBSERVATIONS OF PHYSICAL CHANGES a) b) c) d) e) 1 When the glycine/water mixture was warmed, glycine dissolved after about 20 sec. When the reaction mixture was cooled in ice, white, needle-shaped crystals formed. After filtration and drying of the product, 0. 14 g. f solid was obtained The mp of the crystals was recorded as 205-207 o The solid product was possibly still slightly wet. See the next page for the equivalent flow chart of operations. Of course that's a very simple flow chart. You might want to give it a different design, flow direction, or include pictures. 8 FLOW CHART OF OPERATIONS Take a 5 mL conical vial Add 0. 10 g glycine and 0. 4 mL water Warm slightly to dissolve the glycine Add 0. 3 mL acetic anhydride and stir for about 5 min. Cool the mixture in ice bath for about 10 min. Collect precipitate by suction filtration and wash with a few mL of water.

Spread crystals over a piece of filter paper or watch glass and air dry for 10 min. Record mass and melting point of product. EXAMPLE 4: RESULTS, DISCUSSION, AND CONCLUSIONS In this example, impure acetanilide was

purified by crystallization. The percent recovery and melting point were recorded. RESULTS, DISCUSSION, AND CONCLUSIONS The crystallization of acetanilide gave a 52 % recovery. Loss of product during the hot filtration accounts in part for the low recovery. The melting point of the crystallized product (110-112 oC) is higher than that of the crude material (105-109 oC), indicating a higher degree of purity.

However, the melting point of the crystallized product is lower than the reported literature value (113-115 oC), indicating that it is not completely pure. Perhaps presence of residual water (crystallizing solvent) and error in the melting point determination can account for this. The conclusion is that the crystallized material was in fact acetanilide, based on its melting point, but it was not highly pure. A second crystallization should produce a fairly pure material, but it would also decrease the % recovery.