Macroscale and Microscale Organic Experiments 7th edition Williamson Solutions Manual

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CHAPTER 2 Laboratory Safety, Courtesy, and Waste Disposal

Although microscale experimentation greatly reduces dangers from fire, explosion, and exposure to toxic, carcinogenic, teratogenic, poisonous, and corrosive substances, there are still hazards with which to contend in the organic laboratory. Even on a microscale, eye safety is a prime concern. Students must be required to wear safety goggles (chemical splash goggles) at all times, and the use of contact lenses in the laboratory should be forbidden.

The instructor should read this chapter carefully, noting where local practices differ from the recommendations made in the text, for example how to dispose of a drying agent wet with solvent. These differences should be pointed out to the students, who should then read this chapter and be prepared to answer questions about it. This chapter reflects the latest recommendations of the American Chemical Society (American Chemical Society Joint Board-Council Committee on Chemical Safety, *Safety in Academic Chemistry Laboratories, Vol. 1: Accident Prevention for College and University Students,* 7th ed. Washington D.C.: American Chemical Society, 2003) and the National Research Council of the National Academy of Sciences in their book entitled *Prudent Practices in the Laboratory: Handling and Disposal of Chemicals*, (National Academy Press, Washington, DC, 1995). The most important points of the chapter are emphasized in the Prelab Exercise. It is the instructor's responsibility to see that the safety equipment (eyewash stations, showers, fire extinguishers, etc.) is in place and working properly, that the hoods are operating, and that proper chemical waste containers are provided and labeled. It is very important that waste containers be labeled "Hazardous Waste." A label such as "Waste Solvent" or "Laboratory Waste" does not meet the legal requirements.

The tragic death of Prof. Karen Wetterhaln of Dartmouth College from dimethyl mercury poisoning underscores the necessity for wearing the exactly correct protective gloves (of the 25 types sold) when handling toxic and corrosive chemicals. We continue to agree with the statement made by Williamson in previous editions of this book: "It is probably safer not to wear gloves and immediately wash your hands with soap and water after accidental contact with any harmful reagent or solvent than to wear inappropriate or defective gloves."

The instructor and laboratory manager should be aware of leaving old bottles of material on the shelves gathering dust. This is labeled by the EPA as "inherently waste-like" and old bottles must be disposed of properly.

Answers to Questions

1. $R–O–O–R + 2I^- + 2H^+ \rightarrow 2R—OH + I_2$

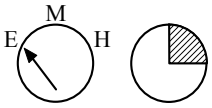
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The orange or brown color comes from dissolved iodine.

2. At ordinary temperatures, all gases except hydrogen and helium cool upon expansion without the production of work (the Joule-Thomson effect). The white solid seen coming from a CO_2 fire extinguisher is dry ice (solid carbon dioxide).
3. Organic solvents float on water and will continue to burn.
4. The safety issues include: (a) not carrying out a reaction in a hood; (b) not taking off the gloves immediately after spilling ether on his gloved hand; (c) pouring ether down the drain instead of putting it in the nonhalogenated organic waste container.

CHAPTER 3 Melting Points and Boiling Points



The five concepts for predicting physical properties of organic molecules are discussed in a detailed consideration of intermolecular attractive forces, a knowledge of which will allow students to predict boiling points, melting points or solubility of one structure relative to another.

If possible, provide the students with a commercial melting-point apparatus. The authors have found that one Thomas-Hoover *Uni-Melt* apparatus for every six to eight students is more than adequate. This apparatus is equipped with a motor-driven stirrer and a periscope device for reading the thermometer. We have found it unnecessary to purchase the thermometer lighting system; room light is adequate. The *Mel-Temp* apparatus is also highly regarded.

The experiments on melting points, recrystallization (Chapter 4), and extraction (Chapter 7) can be run concurrently. These can be finished in about four periods. It is, of course, particularly important for the student to acquire good technique at the outset for the frequently performed operations of crystallization and extraction.

Four methods for the determination of boiling points are discussed in order of their accuracy and experimental difficulty. If enough material is at hand, distillation will give the best results. The next easiest method is to use 0.2 mL of liquid in a reaction tube and then to use one drop of liquid in a 3- to 5-mm diameter tube. The use of a digital thermometer with a surface probe as described on pp. 58–59 of the text is *highly* recommended.

Answers to Questions

1. Depending on the mode of circulation, the observed melting point could be either higher or lower than the true melting point.

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