Introduction to the Organic Laboratory

1 Give reasons for the following safety rules.

(a) Contact lenses should not be worn in the laboratory.
(b) A chemical spill on the skin should be washed off with water, not with solvent.
(c) Solvents are not to be poured down the sink.
(d) Water should not be used to extinguish laboratory fires.
(e) To dilute concentrated sulfuric acid, we pour it onto ice instead of simply mixing it with water.
(f) Broken glassware should be picked up immediately and put in the designated container.
(g) Closed-toe shoes should be worn to laboratory.

answers

(a) Contact lenses can trap chemicals. They may require time to be removed when the eye must be washed. In addition, soft contacts can actually absorb chemicals from the atmosphere in the laboratory and damage the eye.
(b) A solvent may carry the chemical through the barrier of the skin into the system—that is, cause dermal absorption that would not have taken place otherwise.
(c) Solvent fumes in a drainage trough are a fire hazard.
(d) Most organic compounds float on water; therefore, water will cause the fire to speread. Some chemicals, such as sodium metal and metal hydrides, react explosively with water.
(e) When concentrated sulfuric acid is mixed with water, sufficient heat is generated that the acid-water mixture will boil and splatter. The ice cools the mixture sufficiently that this does not occur.
(f) Broken glassware is a hazard which must be removed immediately because another lab occupant may not notice it and inadvertantly cut themselves on it.
(g) Closed-toe shoes protect the feet from both chemical spills and broken glassware.

2 What should you do in each of the following circumstances?

(a) Your neighbor splashes a chemical into his or her eye.
(b) A strong acid spills onto your hands.
(c) You spill a large amount of diethyl ether on the bench top.
(d) Your neighbor's clothing catches fire.
(e) Your reaction flask catches fire.

answers

(a) Hold the lids open and wash the eye with a gentle stream of water for about 15 minutes. It may be necessary to forcibly restrain the individual to do this. If necessary, get help to force the individual to the floor, hold his or her lids open and pour a gentle stream of water over the eyeball.
(b) Immediately, wash your hands under cool running water.
(c) First, tell the instructor and ask for assistance. Ether is highly volatile and flammable. If there are flames in the lab, evacuate immediately. Otherwise, cover the spill with a spill pillow (rags, paper towels) to soak up as much of the spill as possible. Place the soaked spill pillow in the fume hood. Be aware of the extreme fire danger while the fumes are in the air. Know where the fire extinguisher is.
(d) If near the shower, push the neighbor in and turn on the water. If not near a shower, force the neighbor to roll on the floor while another individual gets a fire blanket. Roll the per-

son on the floor while wrapped in the blanket. Prevent the individual from running and fanning the flames.

(e) Inform the instructor and leave the area. Don't scream FIRE and run.

3 Find the following items in your student laboratory:

(a) Fire extinguishers.
(b) Safety wash or eye wash.
(c) EEP (Emergency Evacuation Plan).
(d) MSDS's.
(e) First aid kit.
(f) Closest phone for use in emergency situations.

answers

a-f: The answers to these will be different for each institution.

4 Why is it important to clean up any chemical that you spill in the laboratory? If you do not know the identity of the chemical, where should you put it?

answer

Chemical spills create hazardous waste which must be properly disposed according the rules of the locality as set by university departments and state and local municipalities. If spills are not cleaned up, they might harm the next person to use the lab room. If you do not know the identity of a spilled chemical, ask the instructor where it should be placed for proper disposal.

5 Find the hazard information for each of the following compounds from three different sources, including one MSDS, one printed source, and one Web or online source. Compare and contrast the relative dangers of working with each chemical.

(a) Diethyl ether
(b) Benzene
(c) Methylene chloride
(d) Ethanol
(e) Benzophenone
(f) Sodium chloride

answers

The purpose of this question is to get students looking up hazard information and finding which sources have which type of information. Of course, it will not be possible to check to see if they looked it up in three different sources unless the instructor has them print out the data for each. The answers below are the key hazards of working with each compound.

(a) Diethyl ether is low-boiling and extremely flammable; a radiator, hot plate, or steam line can ignite its fumes.
(b) Benzene is a known carcinogen; it is also volatile, flammable, and toxic.
(c) Methylene chloride has the advantage that it is not flammable. It is toxic and a suspected but not proven carcinogen.
(d) Ethanol is flammable.
(e) Benzophenone is rated as a mild health hazard.
(f) Sodium chloride is non-toxic (except in very high doses).

6 Look up the physical properties of the following compounds:
(a) 1-Bromopentane
(b) Methanol
(c) Sodium bromide

answers

	MW	*bp*	*mp*	*density*
1-bromopentane	151.05	129.6°	-87.9	1.218
methanol	32.04	65°	-93.9	0.791
sodium bromide	102.9	1390	747	3.203

7 Make the following conversions.5.0 g $CH_3CH_2CH_2CH_2CH_2Br$ to moles
(d) 0.100 mol CH_3OH to grams
(e) 2.50 mol NaBr to grams
(f) 10.0 mL concd H_2SO_4 (96%, density 1.84) to moles *
(g) 0.30 mol H_2SO_4 to mL of 6N H_2SO_4

answers
(a) number of moles = (5.0 g) / (mole / 151.05 g) = 0.033 mole
(b) 0.100 mole (32.04 g / mole) = 3.2 g
(c) 2.5 mole (102.9 g / mole) = 257 g
(d) total grams = 10.0 mL x 1.84 g / mL
grams sulfuric acid = 0.96 x 18.4 g = 17.7
moles sulfuric acid = (17.7 g) / (98.1 g / mole) = 0.18 mole
(e) equivalents of sulfuric acid = 0.30 mole x 2.0 eq / mole = 0.60 mole
volume = eq / N = (0.60 eq) (6.0 eq / L) = 0.100 L, or 100 mL

8 You need 45 mL of a 5% aqueous solution of $NaHCO_3$.
(a) What weight of $NaHCO_3$ is required?
(b) How much water will you add?

answers
(a) 45 mL (5 g / 100 mL) = 2.25 g
(b) enough to make the final volume 45 mL

9 What volume of 50% NaOH is needed to prepare 25 mL of 2.0% NaOH?

answers
(a) 25 mL x (2.0 g / 100 mL) = 0.50 g
X mL x (50 g / 100 mL) = 0.50 g, *X* = 1 mL

10 Calculate the percent yield when:
(a) the theoretical yield of a product is 15.3 g and a student obtains 6.9 g
(b) the theoretical yield is 3.1 g and a student obtains 2.7 g

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* Note: the physical properties of sulfuric acid are inside the front cover of the text.

answers

(a) 6.9 g / 15.3 g = 0.45 = 45%

(b) 2.7 g / 3.1 g = 0.87 = 87%

11 For each of the following reactions, (1) identify the limiting reagent, and (2) calculate the theoretical yield of the organic product. (Note: The equations as shown are not necessarily balanced.)

(a) $CH_3CO_2H + NaOH \longrightarrow CH_3CO_2Na + H_2O$

25.0 g 10.0 g

(b) $H_2NCH_2CH_2CH_2NH_2 + HCl\ (12\ M) \longrightarrow Cl^-H_3\overset{+}{N}CH_2CH_2CH_2\overset{+}{N}H_3Cl^-$

5.0 g 10.0 mL

answers

(a) moles of acetic acid = (25.0 g) / (60 g / mol) = 0.42
moles of socium hydroxide = (10.0 g) / (40.0 g / mol) = 0.25
therefore, sodium hydroxide is the limiting reagent
theoretical yield = 0.25 mol x 82 g / mol = 20.5 g

(b) moles of 1,3 diaminopropane = 0.067
moles of hydrochloric acid = 0.12. Since, 2 moles of hydrochloric acid are required per mole of 1,3-diaminopropane, the hydrochloric acid is the limiting agent at 0.060 mol. Therefore, only 0.060 mol of product can be formed.
theoretical yield = 0.060 mol x 146.1 g / mol = 8.8 g

12 What would be the heat source (or heat sources) of choice for boiling each of the following solvents?

(a) Diethyl ether, $(CH_3CH_2)_2O$, bp 35°

(b) Water, bp 100°

(c) Ethanol, CH_3CH_2OH, bp 78°

(d) Acetone, $(CH_3)_2C{=}O$, bp 56°

answers

(a) low boiling and flammable: steam bath, a low-power hot plate or heating mantle

(b) high boiling and nonflammable: hot plate or heating mantle

(c) moderately high boiling and flammable: hot plate, steam bath or heating mantle

(d) low boiling and flammable: steam bath, hot plate, or heating mantle

13 Many student laboratories employ heating mantles for heating reaction mixtures. These devices should not be plugged directly into the electrical outlet. Why?

answer

Heating mantles are not designed to operate at 110 volts. Instead, the mantle is plugged into a variable transformer which is used to adjust the voltage and thus the temperature.

14 You open your laboratory drawer to prepare your glassware for an experiment. You find that your round-bottom flask is attached so firmly to your distillation adapter that you cannot take them apart.

(a) What should you have done when storing your glassware during the previous lab period?

(b) How can you get the two pieces of glassware apart?

answers

(a) Taken it apart.

(b) Hot water, a gentle rap on the bench top.

15 At the end of a reaction, your glassware is covered with a tarry substance. If you use acetone to clean the tar from the glassware, can you place the glassware immediately in a drying oven? Why or why not?

answer

Glassware freshly rinsed with acetone must not be put in a drying oven because the acetone fumes are flammable.

16 You have a summer job as a laboratory technician at a local chemical manufacturing company. What information about safety plans and the chemicals you will be working with should your company or supervisor provide?

The company should have a Chemical Hygiene Plan. This plan should outline how the chemicals in the laboratory are handled. A list of all chemicals should be available to you, as well as an MSDS for each one.