Chem 235 Final Prep

Snappers:

1. What is the definition of a b.p.?
   a. T. at which total vapour pressure = atmospheric pressure.
2. What happens to the boiling point of a mixture as the distillation proceeds?
   a. It will vary as the composition of the liquid being distilled changes (as some distillate is taken out)
3. Why would azeotropes form?
   a. Strong intermolecular forces – e.g. H-bonding.
4. In a simple distillation, the observed boiling points are always…?
   a. Between the boiling points of the pure components, rising gradually as the distillation proceeds.
5. Can a clear cut separation of a binary mixture with a simple distillation be obtained?
   a. No! Only an enrichment of 1 is possible.
6. How can a fractional distillation obtain nearly pure distillates?
   a. As the liquid boils, before the vapour is removed, it is liquefied and revaporized (at a new boiling point). This creates a "zig zag" on the composition curve and allows for obtaining purer compounds.
7. In terms of setup, how does a simple distillation differ from a fractional distillation?
   a. A fractionating column is used in fractional distillation, which permits the repeated exchange of molecules between the liquid and vapour phase. The column is packed with round glass beads for large s.a. to cool the gas.
8. What is a refractive index?
   a. A measure of the change in the light path (refraction) as light passes at an angle from 1 medium to another as a result of different light velocity in the 2 media. It is ADDITIVE.
9. What is a stillhead adapter? What is it used for?
   a. As stillhead adapter with a rubber adaptor and thermometer is used in distillations to connect the RB flask with the west condenser. It guides the condensate to the alternate vessel.
10. What is a takeoff adapter? What is it used for?
    a. Take off adaptor is used as a "spout" for the condensate out of the west condenser. It guides the liquid into the receiver (usually test tube).
11. What is a thermowell?
    a. An apparatus excellent at heating round bottom flasks due to its shape.
12. Why is it important to calibrate a thermometer? How is it calibrated?
    a. To ensure its readings and the subsequent data collected with it is accurate. It is calibrated by putting it in an ice bath for several minutes with the IMMERSION LINE fully submerged and ensuring that the reading on the thermometer is 0 degrees.
13. What is the direction of water flow in a condenser?
    a. Against gravity.
14. What is the heating mantle?

   a. Something used to heat the distilling flask (RB) used in a distillation. Is usually a thermowell.

15. What is a “good” rate for a distillation?
   a. 1 drop off the end of the takeoff adapter every second.
16. Why should the receiver be cooled?
   a. To prevent evaporation of the distillate.
17. Why is operating a distillation adiabatically desirable? How can this be attained? When is this particularly important?
   a. To maintain the equilibrium between liquid and vapour. Can be attained by insulating the distilling flask and stillhead adapter (and fractionating column) with aluminum foil. This is particularly important for fractional distillations.
18. When using an Abbe Refractometer to determine the refractive index of a liquid, how much sample should be used?
   a. Depends on the volatility of the liquid. If non-volatile, 2-3 drops is enough. If it is volatile, used lots (as much as possible).
19. On an Abbe Refractometer, how many significant figures should be read?
   a. 5 (4 decimal places).
20. When is Thin Layer Chromatograpy used?
   a. To quickly separate and qualitatively analyze small amounts of material.
21. What can TLC be used for in an organic lab?
   a. Determine number of compounds in a mixture (number of dots)
   b. Establish if 2 compounds are identical (same Rf values with at least 2 developing solvents)
   c. Appropriate solvent for column chromatography.
   d. Monitor a column chromatographic separation.
   e. Check the effectiveness of separation by column, recrystallization or extraction.
   f. Monitor progress of reaction (a new TLC plate every x period of time)
22. When can TLC not be used?
   a. With volatile compounds.
23. How can a TLC be prepared?
   a. Solid phase (e.g. SiO2 * xH20, Al2O3) spread out on glass or plastic.
   b. Dissolve compound under investigation in volatile solvent and “spot” it on one end of the plates (when solvent evaporates, compound is ADSORBed onto the plate).
   c. Plate is dipped into a shallow pool of developing solvent.
   d. Solvent moves up plate by capillary action.
   e. Compound will move with the liquid phase (more attraction).
24. Why would different compounds move up to different heights?
   a. Different structures and preference for solvents (ie.e intermolecular forces).
   b. Less polar = further.
25. Are Rf values physical constants?
   a. No!
26. What can be done to prevent tailing or streaking?
   a. Adding some acetic acid to the developing solvent to make it more polar.
27. What are micropipets (organic chem)

    a. Capillary tubes that are heated, pulled, and broken down to the correct sizes: 3-4 inches.
28. How can a developing chamber be constructed?
    a. Place half of a filter paper straight edge down into a 100 mL beaker, leaving 1 inch open for viewing (saturates chapmber with vapours of developing solved and reduc evaporation)
    b. Add developing solvent in.
    c. Cover beaker with a watch glass and let stand for at least 5 minutes.
29. How can spotting solutions be prepared?
    a. Few crystals of compound + 4 to 5 drops of a volatile solvent (~10% solution).
30. What should one be careful of when preparing a TLC plate?
    a. Do not break the surface of the adsorbent material.
    b. Do not touch the area where the compounds will travel through (i.e. between the 2 lines)
31. How big should a “spot” be?
    a. 2 mm.
32. Relative to the solvent level in the developing chamber, how high should the spots be?
    a. Above the solvent line.
33. When should the development of the TLC plate be stopped?
    a. When the top of the solvent front travelled to the pencil line at the top of the TLC plate.
34. How can spots on TLC plates be visualized or detected?
    a. UV light – spots only present when exposed.
    b. Iodine chamber (with few crystals of iodine) – spot will stay for a little while after exposure.
35. What kind of spotting solvent can be used?
    a. Methylene chloride
36. What kind of developing solvent can be used?
    a. 5:1 - methylene chloride: ethyl acetate
    b. 10:1 – petroleum ether: ethyl acetate
37. How should Rf values be calculated?
    a. “Solute front”/”Solvent front”
38. What is melting point analysis useful?
    a. Locate impurities (i.e. pure compounds have very sharp melting points) – causes m.p. depression AND widens the range.
    b. Recrystallized compounds have unchanged m.p.
39. What range should be recorded in a melting point analysis?
    a. Temperature at which the first drop of liquid appears until the entire sample has melted.
40. How can a m.p. analysis determine whether 2 compounds are identical?
    a. If the m.p. of the mixture is undepressed identical
    b. M.p. depression different
41. What is the difference between take the m.p. of a known and unknown compound?
    a. Known compound:

        i. Heat rapidly until 15-20 degrees below literature m.p.
        ii. Increase heat by 2 degrees per minute until the first drop of liquid appears.
        iii. Continue until the entire compound melts.
        iv. With a second sample, repeat.
    b. Unknown compound:
        i. Heat rapidly until 1st drop of liquid appears, record temperature.
        ii. Record temperature for when the entire solid melts.
        iii. With a second sample, follow procedure for the KNOWN m.p.

42. How is a thermometer calibrated with a melting point apparatus?
    a. It is used to measure the temperature of a series of compound with known m.p.s and a calibration equation is obtained. Every thermometer reading must be corrected using the equation.
43. On a Fisher-John Melting Point Apparatus, how should the sample be prepared?
    a. Grind a small sample of the compound.
    b. Place a small sample on a spatula.
    c. Place another microcoverglass on top and sandwich the sample.
    d. Wiggle the “sandwich” until the sample is in a thin film (while still touching each other)
44. How should a sample be prepared for a Mel-Temp Melting Point Apparatus?
    a. Grind small sample of compound.
    b. Push sample into a mound and push end of a capillary tube into the sample (3-4 mm).
    c. Pack the sample by dropping it through a long glass/metal tube. Sample should be 2-3mm in length.
45. Why is it important to prepare samples of the right size for melting point analysis?
    a. Too much □ too long to melt, display artificially large m.p.
    b. Too loose □ difficult to determine when the sample starts to melt.
46. How is the mole ratio determined?
    a. Mol % A = [(# moles of A)/(# moles of A + # moles of B)] * 100%
47. What is a temperature lag?
    a. The time between when the heated sample reaches a certain temperature and the time the thermometer displays that temperature. Is a bigger issue with faster heating.
48. Why are recrystallizations performed?
    a. To isolate and purify a substance.
49. When is a solvent pair used?
    a. When 1 single solvent does not fulfill all of the conditions required of a good solvent.
50. Why is depth necessary in a recrystallization?
    a. The crystals need space to grow.
51. Why is a beaker not a good vessel for recrystallization?
    a. High surface evaporation.
    b. Cannot be corked or stopper so that liquid and vapour can equilibriate.
    c. Cannot be swirled to facilitate the dissolving of the solid.
52. Why should an Erlenmeyer flask with ground glass joint be used?
    a. It is designed to be used with a west condenser (connects well).
53. Why should a west condenser be clamped loosely at the middle?

    a. So the Erlenmeyer flask can be swirled.

54. Why is swirling the Erlenmeyer flask important during a recrystallization?
    a. To dissolve the solid.
    b. To avoid bumping.
55. During a hot filtration, why is it important to warm the glassware (Erlenmeyer flask and glass funnel) and add a little excess solvent?
    a. To avoid premature precipitation of the sold on the funnel and receiver.
56. Why is Norit used? How does it work?
    a. To remove coloured soluble impurities. Impurities attach to the surface of the norit by adsorption.
57. In which solvents do Norit work the best? The worst?
    a. Alcohol and aqueous. Least well in hydrocarbons.
58. How much Norit should be added?
    a. Enough to cover about 0.5 cm tip of the spatula.
59. Why must a solution be cooled before adding Norit?
    a. Adding Norit to warm liquid causes uncontrollable frothing.
60. Why is Celite necessary?
    a. Norit is absorbed into the celite’s surface and prevents Norit from going through the filter paper.
61. What are the advantages of allowing crystals to form at room temperature?
    a. Better crystals (structurally) but lower yield.
62. What are the advantages and disadvantages of allowing crystals to form in an ice bath?
    a. Higher yield and shorter crystallization time, but smaller crystals.
63. How can one tell when the recrystallization is complete?
    a. The crystals and the mother liquor are independent of each other.
64. What is a water pump?
    a. A pump that creates suction by using water flow through a sink.
65. In what order should the suction apparatus be turned off?
    a. First pull out the tubing at the water pump
    b. Then shutting off the pump.
66. Why is it important to pull the tubing out of the pump before shutting it off?
    a. To prevent backflow into the pump and the contamination of the sample.
67. What can be done if crystals do not form?
    a. Scratching, seeding, putting in ice bath, evaporate off some liquid, adding a small amount of a liquid in which the solid does not dissolve.
68. On what principle is liquid-liquid extraction based?
    a. Differences in solubility of the components of 2 miscible solvents.
69. Why are successive extractions performed?
    a. To extract the utmost amount of the compound – 1 extraction often leaves things behind because the compound can still dissolve in the first solvent.
70. What is the coefficient of extraction?
    a. The ability of 1 compound to dissolve in 1 solvent compared to another.
71. Why should a separatory funnel never be laid on its side?
    a. It could roll off the bench or have something dropped on it.

72. What is the maximum volume a solution should occupy in the funnel? Why?
    a. 2/3. It's hard to achieve proper mixing otherwise.
73. How should the bottom layer be removed?
    a. Through the stop cock.
74. How should the top layer be removed?
    a. Poured out through the top of the funnel.
75. What is the second solvent called?
    a. Extracting medium.
76. How much extracting medium should be used?
    a. About same volume as the other solvent (divided up into portions)
77. What is an emulsion?
    a. Foamy layer between the 2 solvents. Is a finely dispersed suspension of 1 liquid layer in another as a result of the mutual solubility of the 2 layers increasing.
78. What are drying agents?
    a. Inorganic salts that can form hydrates.
79. How long should one leave a drying agent to dry?
    a. At least 10 minutes.
80. How can a drying agent be removed?
    a. Gravity filtration.
81. In a liquid-liquid extraction, why would we wish to form the salt of a base or acid with the portion in the aqueous solution? What do we do with this salt?
    a. This salt of the base or acid is first changed back to the acid or base (by acid/base reactions) and then successively extracted with ether (or another easily evaporated compound) so that the acidic or basic compound can be isolated out.
82. Should a warm solution ever be extract? Why or why not?
    a. No, extracting warm solutions is dangerous because of the risk of inflammation, combustion, or explosion.
83. How is the partition coefficient defined?
    a. K = C2/C1, where C2 and C1 are equilibrium concentrations of solute A in solvent 2 and solvent 1 respectively.
84. In a solubility test of a mixture for liquid-liquid extraction, how much solvent should be used in the test? The solute?
    a. A few crystals of the solute in 2-3 mL of solvent.
85. How can solubility be increased in solubility test for liquid-liquid extraction?
    a. By agitating the test tube or heating it.
86. How much solvent and solute should be used for a solubility test for recrystallization?
    a. A few crystals of solute in 3 drops of solvent.
87. Describe the process of a test for solubility for recrystalization.
    a. 3 drops solvent + few crystals of solid.
    b. If compound does not dissolve, place in a boiling water bath for 1 minute.
    c. If compound does not dissolve, add up to 20 drops of the solvent slowly until a maximum of 20 drops. Stop after 2 minutes of boiling if dissolving has not occurred.
88. Describe the process for a solvent pair recrystallization.

   a. Dissolve compound in a minimum amount of the boiling solvent in which it is very soluble (5-10 mL).
   b. After the solution boils and completely dissolves, add a small amount of the solvent in which the compound is insoluble until a trace of cloudiness appears and does not disappear upon swirling or continued heating.
   c. Add enough of the first (soluble) solvent until the cloudiness just disappears, then set aside to crystallize.

89. How do impurities affect melting points?
   a. Causes the range to widen and usually to depress.
90. Why are esters usually synthesized in the lab?
   a. Difficult or expensive to isolate them from natural organisms.
91. What does isoamyl acetate smell like?
   a. Bananas
92. How is isoamyl acetate obtained?
   a. Acetic Acid + isoamyl alcohol.
93. How can the product of ester synthesis, isoamyl acetate, be encouraged?
   a. Excess acetic acid (le chatelier’s principle).
94. In ester synthesis, which layer should be kept? Organic or aqueous? Top or bottom?
   a. Organic, top.
95. In the synthesis of isoamyl acetate, what part of the distillate should be collected (between which temperatures)?
   a. Fraction between 130 and 140 degrees.
96. A mixture of compound A (lower b.p.) and compound B (higher b.p.) are distilled in a simple distillation. Between the 10th and 30th fractions, which one is more enriched in compound A? Compound B?
   a. 10th Compound A
   b. 30th Compound B
97. Compound A (lower b.p.) and compound B (higher b.p.) are distilled in a fractional distillation. Which compound is mostly present in the distillate?
   a. Compound A.
98. While refluxing with an RB flask, thermowell, and west condenser, where should the clamp be placed?
   a. On the mouth of the RB flask.
99. What kind of heat source should be used to reflux a volatile substance? A non-volatile substance?
   a. Volatile hot water beaker
   b. Non-volatile thermowell
100. In a Grignard reaction, why does everything (glassware and reagents) be dry?
   a. Water will cause the formation of an alkane and HOMgX, which are undesirable products.
101. Why would byproducts such as biphenyl form?
   a. The synthesized Grignard reagent will react with the R-X used to synthesize the Grignard reagent. These reactions are catalyzed by light or darkness.
102. Why is a drying tube used for Grignard reactions?

    a. To keep out atmospheric moisture.

103. Where should the clamp be on the condenser in a Grignard reaction?
    a. The clamp should not be on the condenser! It should be at the neck of the RB flask!

104. What kind of heat source should be used for refluxing in a Grignard reaction?
    a. Hot water bath.

105. In a Grignard reaction, what is used as a catalyst?
    a. Iodine.

106. Describe the process used to start the Grignard reaction. What are the major indicators that the reaction is going well?
    a. Add an iodine crystal into the RB flask.
    b. Warm the rub flask with one's hands.
    c. Shake/swirl the flask vigorously while holding onto the drying tube until:
        i. Bubbling from the magnesium metal (not solvent)
        ii. Appearance of precipitate/cloudiness
        iii. Heat from the bottom of the flask
        iv. Disappearance and the reappearance of red/brown iodine colour

107. Why is it important to place the RB flask in an ice bath after the Grignard reaction is initialized?
    a. To temporarily suspend the reaction to set up the condenser and add the anhydrous diethyl ether.

108. Why is it important to add acid and ice to intermediate ("triphenylmethanol ion") in the fume hood?
    a. Build-up of H2 gas, which could be flammable.

List Questions:

109. What is distillation used for? (2)
    a. Separating mixtures with different boiling points
    b. Purifying a liquid

110. Under what conditions should the distillation technique be used? (3)
    a. At least 1 component of the mixture must be volatile
    b. If more than 1 volatile liquid, their b.p.s must be separated by at least 50 degrees.
    c. Components must not form an azeotrope.

111. What does refractive index depend on? (3)
    a. Wavelength of light
    b. Temperature
    c. Relative concentrations of the solution/liquid

112. What are the benefits of TLC?
    a. Fast
    b. Reproducible
    c. Uses small amount of material (1-100 microgram)

113. What are the basic requirements of a good solvent for recrystallization? (4)

- a. Differentially dissolve the compound (poorly at room temperature, very well at its boiling point) in a reasonable volume (i.e. no more than 10 to 20X the weight of the solid).
- b. Cannot react chemically with the compound to be purified.
- c. Should either dissolve impurities exceedingly well (so that they remain in solution even when it is cooled) or not a all (so they can be filtered out).
- d. Should have a boiling point which is much less than the melting point of the solid to avoid “oiling out” (i.e. solid melts instead of dissolves).

114. Why is an Erlenmeyer flask good for recrystallization? (5)
    - a. Narrow neck allows for refluxing so that surface evaporation is minimized.
    - b. Can be corked so that vapour and liquid can equilibrate during crystallization.
    - c. Can be swirled to facilitate the dissolving of the solid.
    - d. It is relatively easy to remove crystals from an Erlenmeyer flask.
115. In a liquid-liquid extraction, what conditions must the 2 solvents satisfy?
    - a. One solvent completely dissolves both compounds in the mixture (or if 1 is suspended)
    - b. The other solvent must be immiscible with the first solvent.
    - c. The other solvent must dissolve only 1 component of the original mixture.
116. What are the components of a separatory funnel?
    - a. Plug, white washer, black O ring, lock nut, funnel.
117. How can an emulsion be removed?
    - a. Waiting for 10 to 20 minutes.
    - b. Swirling the flask gently.
    - c. Add concentrated NaCl (if water is used)
118. List some common drying agents.
    - a. Magnesium sulphate
    - b. Sodium sulphate
    - c. Calcium chloride
119. How can organic solvents from the desired compound be removed?
    - a. Evaporation over heat, at reduced pressure, or in a rotary evaporator.
    - b. Distillation
120. What information should be placed on a proper label?
    - a. Experimenter’s name; lab section; locker number
    - b. Date sample was prepared
    - c. Name and structure of the compound
    - d. Weight or yield (e.g. percentage yield) of the sample
    - e. Any measured physical constants (NOT literature value) – e.g. m.p., b.p., refractive index, density (NOT Rf value!)