

Available Points: 50

- 1. Describe Size Exclusion Chromatography. (4 points)**
- 2. Describe osmotic pressure. (3 points)**
- 3. List 2 advantages and 2 disadvantages of osmotic pressure. (4 points)**
- 4. What are the three advantages of FT-IR? Briefly describe each advantage. (6 points)**
- 5. Which two characterization techniques can be used to determine T_g ? (2 points)**

Multiple Choice. Choose the answer that is NOT true for each technique. (6 points)

6. Dynamic mechanical spectroscopy (DMA)
 - a. It can measure viscosity
 - b. It is a thermomechanical technique
 - c. It measures viscoelastic properties
 - d. It can measure the strength of a solid polymer
7. Size exclusion chromatography (SEC)
 - a. It uses a porous polystyrene gel
 - b. The molecular size is separated based on the chemical interaction
 - c. The larger molecules come out of the column earlier than the small ones
 - d. It is a relative molecular weight measurement technique
8. Thermogravimetric analysis (TGA)
 - a. Heat flux relative to the reference material is measured
 - b. Weight loss as a function of the temperature forms the basis for the thermogram
 - c. Char yield is one of the important quantities measured
 - d. Crystallization, melting, and T_g are the quantity that cannot be measured
9. Differential scanning calorimetry (DSC)
 - a. It is a technique that can study thermal events such as liquid crystalline transitions
 - b. Crystallization and melting show an endothermic peak whereas T_g shows an exothermic peak
 - c. Only a few mg of sample is sufficient for the measurement
 - d. Conversion of a thermosetting polymer, such as epoxy, can be studied
10. Nuclear magnetic resonance spectroscopy (NMR)
 - a. ^1H NMR has much higher sensitivity than ^{13}C NMR in part due to the near 100% of the natural abundance of the ^1H atoms
 - b. NMR is one of the most useful techniques to identify the number of components in an unknown sample
 - c. The sensitivity of the technique increases as the strength of the main magnet used increases
 - d. NMR, IR and elemental analysis are the three typical methods that are most frequently used for molecular structural identification
11. Fourier transform infrared spectroscopy (FT-IR)
 - a. Infrared radiation absorbed corresponds to the vibrational energy level
 - b. The energy source emits a single frequency radiation like a laser
 - c. FT-IR provides higher signal-to-noise ratio than the traditional dispersive IR instruments
 - d. Conne's and Fellgets' advantages are a few of the unique advantages of FT-IR

12. Draw a DSC thermogram from a semi-crystalline material. Label T_g , T_m , and T_c . Don't forget your axis labels. (4 points)

13. Choose an appropriate characterization technique from the list below that matches the situation. (8 points)

- Size exclusion chromatography (SEC)
 - Nuclear magnetic resonance (NMR)
 - Dynamic mechanical spectroscopy (DMA)
 - Thermogravimetric analysis (TGA)
 - Fourier Transform Infrared spectroscopy (FT-IR)
 - Differential scanning calorimetry (DSC)
 - Matrix-assisted laser desorption/ionization time of flight mass spectrometry (MALDI-TOF MS)
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- a. Detection of a natural polymer that was produced in different locations of the world. It is known that the isotope ratio of those locations are different and the natural polymer incorporate those isotopes.
 - b. Studying the effect of quenching on the degree of crystallinity change
 - c. Verifying if a polymer is a cross-linked material or a high molecular weight thermoplastic
 - d. Studying if a carbonyl containing polymer is hydrogen bonding to a hydroxyl containing polymer

- e. Determining if an unknown polymer is a polymer blend of homopolymers derived from monomer X and Y or a copolymer consisting of the comonomers XY
- f. Investigating the tacticity ratio of a polymer to see if the polymer is isotactic rich or syndiotactic rich
- g. To compare which polymer is more fire-safe
- h. Gelation point upon polymerization of a benzoxazine resin

14. Fill in the blanks in the following paragraph with the correct characterization technique using the abbreviations from Problem 13. (5 points)

I have just synthesized a benzoxazine monomer in the laboratory. I first used ____ to confirm the monomer structure because it is the most precise way to determine the structure of my compound. To confirm that polymerization took place, I used ____ multiple times for different temperatures to see when the oxazine ring stretch disappeared. When this stretch disappears the polymer is formed. I used ____ and looked at the char yield to learn more about the fire retardant properties of the polymer. To study the mechanical properties, ____ was used to figure and the T_g and crosslinking density of the polymer were measured. I used the $\tan \delta$ peak to record the T_g . Lastly, I want to know the molecular weight distribution (M_n , M_w , M_z) of my polymer so ____ was used.

True or False (3 points)

- 15. DSC detects heat flux differences between an empty sample pan and a pan with a sample. The advantage of DSC is the ability to measure thermal properties with a very limited amount (mg) of the sample.
- 16. NMR is ideal in determining detailed chemical structures of liquid samples. Using the chemical shift concept, one can readily determine the chemical structure of the component in the unknown sample. NMR can also be used to study molecular mobility using relaxation times such as T_1 and T_2 relaxation.
- 17. DMA is a technique used to measure rheological properties. $\tan \delta$ is often used as a useful method to determine the T_g of a polymer. However, a more precise definition of the T_g uses G'' rather than $\tan \delta$.

18. Draw a DMA spectra for a cross-linked material. Label G' , G'' , and $\tan \delta$. Don't forget your axis labels. (5 points)