

Exam 3
Chem 311
Fall 2004

Each problem is worth 5 points! Do 1-19 and choose between 20 and 21.

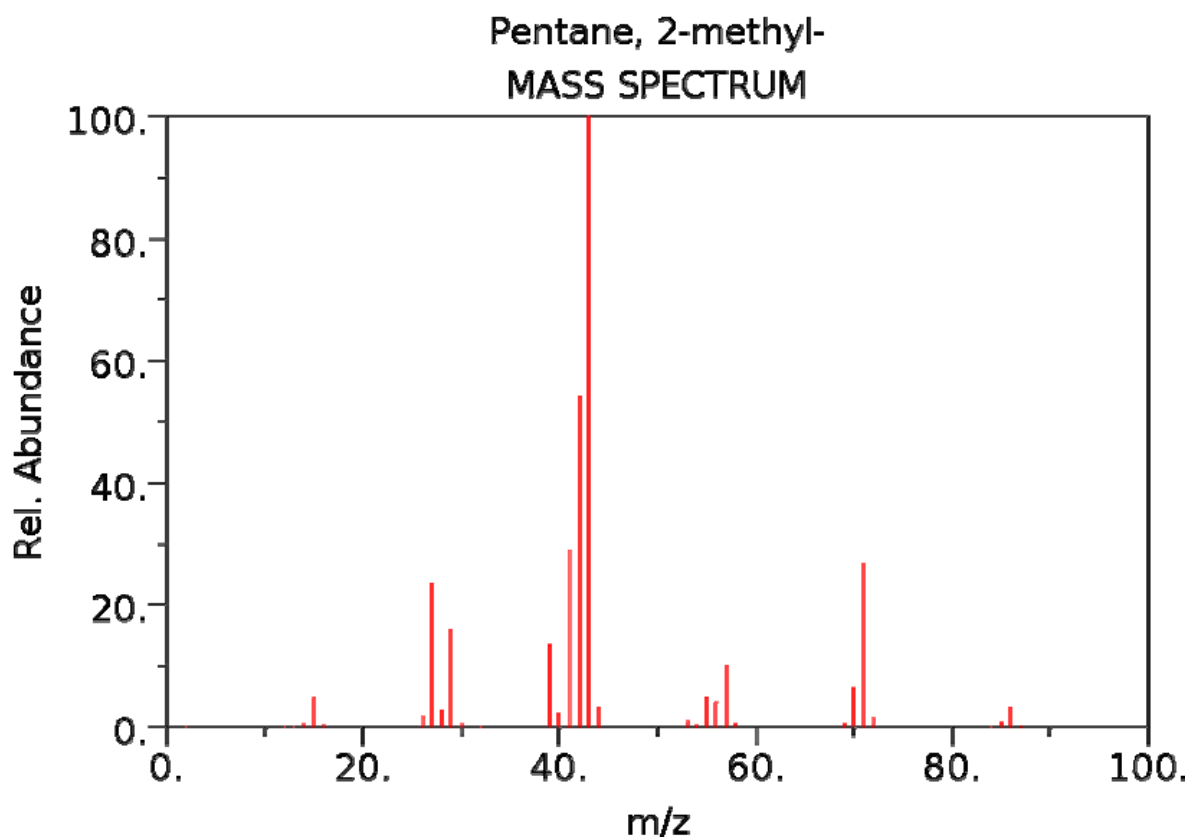
1. Dilute solutions of CuNO_3 are blue because the d-orbital transitions absorb red light. Do these same transitions occur in the analysis of dilute CuNO_3 solutions by ICP-AES? Explain!
2. Why is it necessary to use quartz cuvetts in the UV region, but less expensive glass or plastic cuvetts are acceptable in the visible region? Why are salt (NaCl or KBr) plates used to hold the sample in FT-IR applications?
3. Sketch a diagram of a typical spectrofluorimeter.
4. Why do UV-vis spectra of organic compounds tend to exhibit very broad absorption peaks? You can draw an energy diagram to help explain!
5. In Atomic Absorption experiments each element has its own source (hollow cathode lamp). Why? and what does this mean in terms of selectivity of AA experiments?
6. In theory how many unique IR absorption peak(s) derive from a single vibrational mode in a molecule? How does this relate to functional group analysis?
7. What are the advantages of using Fourier Transform analysis in the IR region?
8. What is the function of the Michelson interferometer and why is it a necessary component of the FT-IR instrument?
9. Why is FT-NMR a relatively insensitive technique? In FT-NMR the bigger the magnet, the better. Why?
10. What is *actually measured* in the FT-NMR experiment?

11. The critical parameters in FT-NMR are chemical shift and spin-spin splitting. Which of these two is the critical parameter that provides information about structural connectivity between atoms?
12. Why can't GC be used to separate involatile mixtures (such as protein digests)?
13. What is the main advantage of using mass spectrometry instead of FID as a GC detectors.
14. Describe the mechanism of ionization in electron impact ionization mass spectrometry.
15. What is the nature of the reverse-phase HPLC packing (stationary phase)?
16. Why is collision-induced decomposition (CID) experiment critical for obtaining structural information in HPLC-MS experiments?
17. The following linear mobile phase gradient was used to fractionate an extract of phospholipids: 0 % B @ 0 min, 100 % B @ 30 min., where A is H₂O and B is MeOH. All of the phospholipids elute between 15 and 20 minutes. Propose an adjustment to the gradient that would be expected to result in a greater degree of fractionation. Draw a plot of %B (y-axis) vs. retention time (x-axis) for your proposed gradient.
18. In FT-IR and FT-NMR applications signal-to-noise ration (S/N) increases with $n^{1/2}$, where n is the number of spectra acquired. If a single FT-IR spectra has a S/N ratio = 2, **how** many scans must be averaged (in theory) to obtain FT-IR spectrum with a S/N ratio of 20.

19. For good precision in GC-MS applications (errors of less than 1%) it is necessary that the chromatographic peaks are well-defined by at least 15 points across the chromatographic peak.

A 30.0 min temperature program GC-MS experiment was performed on gasoline. 2-methylpentane eluted at 9.62 min with a peak width of 10.0 seconds. **What** is the minimum mass spectral scan rate required for precise quantitative analysis of 2-methylpentane under these conditions? Using this scan rate, **how** many mass spectra would be collected throughout the entire 30.0 minute GC-MS run? **Is** the molecular ion of 2-methylpentane present in its EI spectrum given below? The ion of greatest intensity is at m/z 43.

Propose a structure for the fragment.

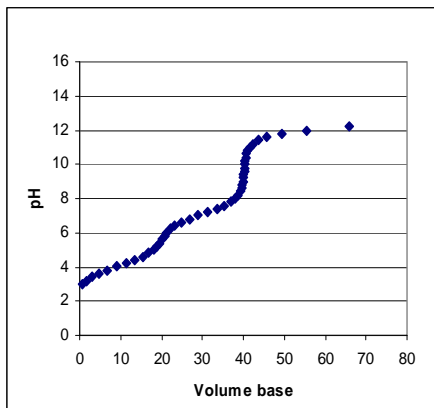


NIST Chemistry WebBook (<http://webbook.nist.gov/chemistry>)

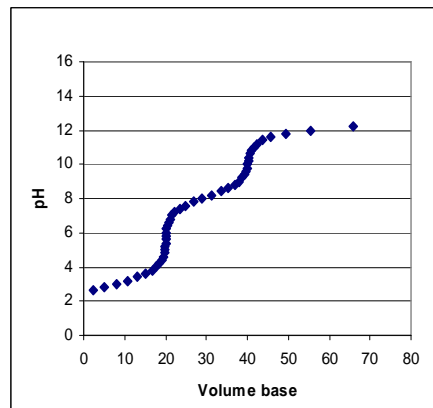
20. 100.00 ml of a 0.0120 M solution of SnCl_2 (soluble salt) in 1 F HCl is titrated with a 0.100 M standard solution of Cerium(IV) in 1 F HCl. Calculate the volume it will take to reach the equivalence point.
21. A 100.00 ml aliquot of a 0.02056 M solution of Threonine-HCl is titrated with 0.1025 M NaOH. Calculate the volume it will take to reach the equivalence point. Which titration curve below best represents this threonine-HCl titration.

Threonine: $\text{pK}_{\text{a}1} = 2.088$ $\text{K}_{\text{a}2} = 9.100$

A



B



C

