Department of Chemistry
Cumulative Examinations

August 23, 2014

You may choose to answer any exam from any area covered in the examination booklet. Each exam may contain multiple parts. You may answer more than one exam but each exam is scored separately and is treated as an individual examination result. Thus, answering parts of two exams with a score of 50% would not yield a 100% grade for this cumulative exam. Instead you would receive 50% on each examination attempted.

This booklet contains five examinations.

1) Analytical Cumulative Examination, Page 1
2) Biochemistry Cumulative Examination, Pages 2-5
3) Inorganic Cumulative Examination, Pages 6-7
4) Organic Cumulative Examination, Page 8
5) Physical Cumulative Examination, Pages 9-10

On your examination booklet:

1) Print your student ID number.
2) Print the Exam Booklet number.
3) Print the question number you are answering.
4) Print the Exam Date.

Do not write your name anywhere on the examination booklet. Each exam will be scored anonymously. If you attempt more than one exam, you must use a separate examination booklet for each examination.

When you complete the examination, return the examination and your answer booklet to the proctor. Exam results will be posted on bulletin board #2B on the north side of the hall near BROWN 2124.
Analytical Cumulative Exam August 2014

Question 1 [100 points]
Consider the following problems in Analytical Chemistry each of which has been adapted broadly from a recently published paper in a leading analytical chemistry journal. In each case indicate what procedure you would select to solve the problem. Start your answer at the most general level, and then proceed to more specifics on the methodology, its physical and chemical basis, the types of results obtained and how they are interpreted. It may be necessary to provide background information on the analyte, the method, etc. You will not be able to be highly specific on every problem but go as far as you can. Avoid vagueness and guesses. The references given are for your information after the exam – it is not necessary to have read specific references to answer the question. The question is intended to elicit rational thinking about analytical chemistry and to allow you to display deep knowledge in the area. The answer must be a coherent piece of writing.....not point form. Language counts as does an organized answer. [You are encouraged to make notes on each answer before writing the graded answer]

ANSWER FIVE of SIX

a) Identify the phosphorylation sites in a peptide

b) Immunoaffinity assay for the flame retardant tetrabromobisphenol A (TBBPA)

c) Determine enantiomeric purity in the product of the following enantioselective reaction performed using a chiral phosphoric acid.

   ![Chemical structure](attachment:image.png)


d) Classify diseased and healthy tissue sections by metabolomics

e) Record chemical images of cholesterol esters in intact tissue using second order microscopy
   Li Li, Haifeng Wang, Ji-Xin Cheng, Biophysical Journal 89 (2005) 3480-3490

f) Determine the trace elements in a meteorite by a laser based method
   Andrew J. Campbell and Munir Humayun, Anal. Chem. (1999), 71, 939-946
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**Question 1.** *(10 Points)*

Draw the peptide CSHADYK at pH 7.4. Don’t worry about stereochemistry for this one.

**Question 2.** *(10 Points)*

Explain the action of an enzyme on a simple Substrate → Product chemical reaction that is thermodynamically favorable. To do this, draw a labeled free energy versus reaction progress diagram. In this diagram use a solid line to show the uncatalyzed reaction trajectory. Use a dashed line to show the catalyzed reaction trajectory.

**Question 3.** *(10 Points)*

In one sentence or less describe:

a) How does an enzyme affect the forward and backward reaction rates?

b) How does an enzyme affect the reaction equilibrium?
**Question 4. (20 Points)**

In analyzing the kinetics for a general enzyme reaction: \[ E + S \overset{k_1}{\underset{k_{-1}}{\rightleftharpoons}} ES \overset{k_2}{\rightarrow} E + P \]

a) Write the Michaelis-Menten equation for the rate of product formation.

b) Explain the Michaelis-Menten assumption about the enzyme-substrate equilibrium.

c) Explain the Briggs-Haldane assumption about the enzyme-substrate complex.

d) As written, explain what implicit assumption is made about the chemistry step in this reaction to simplify analysis (think back to question 3).

e) How does this assumption constrain how you experimentally measure reaction rates?

f) Define the Michaelis constant \( K_M \) in terms of the rate constants above and explain what it is.

g) Explain when \( K_M \) is approximately equal to the enzyme-substrate dissociation constant \( K_0 \).

h) Explain what \( k_{\text{cat}} \) is and be sure to talk about substrate concentration.

i) If you could only know one measurement from someone else’s experiment, explain whether you would want to know \( k_{\text{cat}} \) or \( V_{\text{max}} \) to compare kinetic efficiency of two enzymes.

k) Explain why \( k_{\text{cat}}/K_M \) is more useful than \( k_{\text{cat}} \) as a measure of catalytic efficiency under physiological conditions.

**Question 5. (20 Points)**

A serine protease similar to chymotrypsin uses a catalytic triad of residues H, D, and S to cleave substrates after a tyrosine residue (\( R_1 \) and \( R_2 \) below are the peptide chains adjacent to the tyrosine):

\[ R_1 - Y - R_2 + H_2O \rightarrow R_1 - Y + R_2 \]

a) Explain what type of catalytic mechanism the S residue is involved in.

b) Explain what type of catalytic mechanism the H residue is involved in.

c) Explain what role the D residue plays.

d) Draw a proposed mechanism for the enzyme catalyzed peptide bond hydrolysis.
**Question 6. (10 Points)**

An unknown enzyme hydrolyzes the substrate p-nitrophenylacetic acid.

The reaction progress can be followed by monitoring the absorbance of the nitrophenolate at 400 nm, as shown in the pre-steady state experiment shown below.

a) Explain what the shape of this plot suggests about the complexity of the mechanism.

b) What can pre-steady state experiments determine that steady-state generally cannot?

c) Draw a diagram of a stopped-flow instrument and explain how it is used.

**Question 7. (10 Points)**

Below is the pH rate profile, $k_{cat}/K_M$ vs. pH, for an enzyme exhibiting Michaelis-Menten kinetics.

a) Explain what two amino acids you might expect are in the active site.

b) A different enzyme has a similar pH rate profile, but an active site cysteine has been associated with the left/rising half of the curve. Explain what this suggests about how the active site environment affects the cysteine.
c) In the enzyme in (b), a residue far from the active site has been associated with the right/falling half of the curve. Explain what this might suggest about how this protein regulates its enzyme activity.

**Question 8. (10 Points)**
A kinetic isotope effect (KIE) can be observed as the difference in reaction rates between different isotope-labeled species, usually expressed as a ratio. A primary KIE is observed when the isotope-substituted atom is involved in a bond being formed or broken. For example, C–H bond breakage during hydride transfer from NADH. When either atom is substituted by its isotope, a KIE can result from a change in the vibrational stretching frequency.

a) Given this brief description, explain whether you expect KIE’s for hydrogen-deuterium (k_H/k_D) or hydrogen-tritium (k_H/k_T) substitution to be greater. Explain whether you expect KIE’s for hydrogen-deuterium (k_H/k_D) or ^13^C carbon–^12^C carbon (k_{12C}/k_{13C}) substitution to be greater.

b) A maximum KIE value for k_H/k_D of ~6.9 at 25°C can be approximated by considering the difference in zero point energies (ZPE) of the C–H and C–D bonds. For elements in the first and second periods, ZPEs are the primary contribution to the isotope effect. In the figure below (Fig. 2-9A from Frey and Hegeman, 2007), the transition state is symmetrical, and there is negligible difference between the C–H and C–D transition state energies.

![Diagram of reaction progress]

In the ground state, ZPE = hν/2. From IR spectroscopy, the C–H stretch is at ν ~ 2900 cm^{-1} and the C–D stretch is at ν ~ 2100 cm^{-1}. Derive the max value for k_H/k_D. (Planck’s constant h = 6.626 x 10^{-34} J·s; gas constant R = 8.314 J·K^{-1}·mol^{-1}; Avogadro’s constant N_A = 6.022 x 10^{23} mol^{-1}; c = 3 x 10^{8} m/s)

c) As discussed above, semiclassical transition state theory predicts a maximum KIE value of ~6.9 for k_H/k_D. However, there are examples of enzymes, such as methane monooxygenase, in which KIE values of ~100 have been measured. What quantum mechanical phenomenon could a KIE>>7 suggest about a catalytic mechanism?
Inorganic ChemE – August 23, 2014

Survey of Inorganic Chemistry

Section 1: Electronic Configurations (13 points)

1. Give the ground state electronic configurations beyond the Noble Gas core and the number of unpaired electrons for:

a. Cu  

b. Po  

c. Ni^{3+}  

d. Gd^{2+}  

e. Th^{2+}  

2. Give the ground state term symbols for each of the following free ion electronic configurations:

a. Ni^{4+}  

b. Cr  

c. f^6p^2  

Section 2: Quantum Numbers (10 points)

1. For a “6d” orbital, give the number of radial and planar nodes.

2. How many electrons are in a filled “6h” subshell?

3. Assuming a regular filling order, what orbital will accept the “next” electron after the “5g” subshell is filled in building up the electronic structure of an atom?

4. What are two elements in their +3 oxidation states that possess five unpaired electrons having n = 4 and l = 3?

Section 3: Bonding (26 points)

1. Draw the bonding interaction between a transition metal d-orbital and the appropriate orbitals on NO^+, showing both the σ and π interactions.

2. Upon reduction of the metal to a lower oxidation state, what will happen to the NO^+ bond length? What trend would you expect to observe by infrared spectroscopy?

3. Draw the structure of XeF_5^-, clearly showing the geometry and the positions of any lone pairs. Also, indicate the point group symmetry.

4. Draw the structures showing the mechanism for F exchange in HPF_4 by a Berry pseudorotation mechanism.

5. Consider the following point groups: D_{3d}, T_d, C_{3v}, C_6, C_5. List the point groups in which the molecules can’t be polar.
6. For a square planar metal complex $ML_4$, sketch the sigma antibonding molecular orbital for $d_{x^2-y^2}$.

Section 4: Nomenclature (8 points)

1. Name or provide the formula of, as appropriate, for each of the following:
   a. potassium dioxopentafluorouranate (VI)
   b. cyclopentadienidotetracarbonylniobium(I)
   c. [Cr(NH$_3$)$_2$(OH)$_2$(OH)] (NO$_3$)$_2$
   d. K$_2$[Co(NCS)$_4$]

Section 5: Coordination Chemistry (23 points)

1. Given $[CoF_6]^{3-}$, $\Delta_o = 13,000$ cm$^{-1}$, $E_p = 21,000$ cm$^{-1}$ ($E_p$ = pairing energy)
   a. Is it high or low spin?
   b. How many unpaired electrons does it have? Show your work.
   c. What is the crystal field stabilization energy?
   d. Predict the spin only magnetic moment.

2. How many unpaired electrons are in K$_2$[Fe(CN)$_4$]? Show your work.

3. Which of the following would be expected to undergo a LARGE Jahn-Teller distortion: $t_{2g}^4$, $t_{2g}^6$, $e_g^1$, $t_{2g}^4e_g^2$, $t_{2g}^3e_g^3$? Explain your answer.

Section 6: Solid State Chemistry (20 points)

1. Calculate the lattice energy of AlCl$_3$ (per mole) using the following data:

   $\Delta H_{\text{sub}}$ of [Al (g)] = + 291 kJ / mol
   $\Delta H_{\text{diss}}$ of [Cl$_2$ (g)] = + 243 kJ / mol
   $IE_1$ (Al) = + 578 kJ / mol
   $IE_2$ (Al) = + 1817 kJ / mol
   $IE_3$ (Al) = + 2745 kJ / mol
   $\Delta H_{\text{EA}}$ (Cl) = - 349 kJ / mol
   $\Delta H$ of [AlCl$_3$ (s)] = - 704 kJ / mol

   (IE = ionization energy, EA = electron affinity, sub = sublimation, diss = dissociation)

2. The structure of $\alpha$-tin (Atomic Weight = 118.71) is shown to the right. If the length of an edge of the unit cell is 650 pm (1 pm = 10$^{-12}$ m), calculate the density of the metal.
The following transformations are from recent publications in Organic Letters (2014, 16, ASAP). Provide a reasonable mechanism for any five of the following (5x20 pts.). (Extra credits (EC) are worth 3 pts each; a total of 15 pts).

I. \[R-\text{MgX'} \xrightarrow{\text{L}_n\text{PdCl}_2 \text{(cat.)}} \text{TMEDA, THF, rt}} R'\]
   
   EC: Name this reaction

II. \[\text{m-CPBA} \xrightarrow{\text{EIOH, rt, 0.5 h}} \xrightarrow{\text{then 80 °C, 0.5 h}} \]

   EC: Write the structure of m-CPBA

III. \[\xrightarrow{\text{DMF, microwave}} \]

   EC: Name the key reaction

IV. \[\xrightarrow{\text{CIPh}_3\text{PCH}_2\text{OMe}} \xrightarrow{\text{t-BuOK, THF, rt, 2 h}} \]

   EC: Name the reaction

V. \[\xrightarrow{\text{Cul (10\%)}} \xrightarrow{\text{CH}_2\text{Cl}_2, 40 °C}} \]

VI. \[\xrightarrow{\text{Oxymercuration-adof reaction}} \]

   EC: Provide the reagents necessary for oxymercuration
This course is focused on the bonding, spectroscopy, quantum mechanics, statistical mechanics, and thermodynamics of the prototypical homonuclear diatomic molecule $H_2^+$. 

Potentially helpful equations.

\[ E = mc^2, \quad 1 \text{ gross} = 144 \text{ units}, \quad 1 \text{ acre} = 0.4047 \text{ hectare}. \quad e^{i\pi} = -1. \]

\[ Q = \frac{q}{N_1}; \quad q = \sum g_i e^{ikr}; \quad q_{\text{val}} = q_{\text{ion}} + q_{\text{elec}}; \quad q_{\text{rot}} = \sum J g_i e^{ikr} = \sum J (2J+1) e^{ikr} J(J+1) \]

\[ E_{\text{rot}} = N_k g^2 \left( \frac{\partial \ln q_{\text{val}}}{\partial T} \right); \quad C_V = \left( \frac{\partial E}{\partial T} \right)_V; \quad \text{Rigid Rotor:} \quad \hat{H} = H = \frac{\hbar^2}{2I} \left[ \frac{1}{\sin \theta} \frac{\partial}{\partial \theta} \left( \sin \theta \frac{\partial}{\partial \theta} \right) + \frac{1}{\sin^2 \theta} \frac{\partial^2}{\partial \phi^2} \right] \psi \]

1. In atomic units, the Hamiltonian describing $H_2^+$ is given by the following equation:

\[ \hat{H} = -\frac{1}{2} \nabla^2 - \frac{1}{r_a} - \frac{1}{r_b} + \frac{1}{R} \]

Perturbation theory suggests that the molecular orbitals for $H_2^+$ can be generated from a basis set consisting of linear combinations of atomic orbitals [e.g., $\psi = N(\phi_a + \phi_b)$ for the wavefunction describing the sum of the two hydrogenic 1s orbitals, in which $N$ is a normalization constant].

a. Explain either qualitatively or mathematically the meaning and role of the exchange, correlation, and overlap integrals that collectively combine to generate the potential energy surface for $H_2^+$.

b. What are the driving interactions the result in bond formation?

2. a. Sketch an approximate potential energy surface for $\psi = N(\phi_a + \phi_b)$ and $\psi = N(\phi_a - \phi_b)$ as a function of interatomic separation, labeling the sum and difference.

b. Include an order-of-magnitude estimate for the equilibrium bond length.

c. Sketch isosurfaces (i.e., contours of constant probability density) for the electronic molecular orbitals of $H_2^+$ produced from both the sum and difference of the two 1s orbitals at the equilibrium bond length (hint: $\phi_{1s} = N \psi e^{-\frac{r_a}{\sigma}}$).

3. a. Based on the sketches of the molecular orbitals corresponding to the sum and difference states, is the ground to electronic state electric dipole allowed or forbidden?

b. If it is allowed, in which direction is it polarized and why? If not, why not? Note – the explanation relating the sketches of the molecular orbitals to the electronic transition moment is worth more than the correct result.

4. Explain how you would recover the vibrational constant based on the calculated potential energy surface.
5. Explain how you would recover the rotational constant based on the calculated potential energy surface.

6. The solutions to the Schrödinger Equation for a rigid rotor are the spherical harmonics, which also describe the angular part of the electron motion in the H-atom.
   a. Prove that the following spherical harmonic wavefunction is also an eigenfunction of the angular moment operator; 
      \[ \hat{L}_z = -i\hbar \frac{\partial}{\partial \phi} \].
      \[ \psi(\theta, \phi) = N \sin \theta e^{-i\phi} \]
      b. What are the corresponding eigenvalues?
      c. Based on the eigenvalues, is the wave traveling clockwise or counterclockwise about the z-axis of H₂⁺?
      d. What are the H-nuclei doing during this process?

7. The following wavefunction is also a solution to the Schrödinger equation describing the rotational motion of H₂.
   a. Prove that it is not an eigenfunction of the angular moment operator.
      \[ \psi(\theta, \phi) = N \sin \theta (e^{i\phi} + e^{-i\phi}) = 2N \sin \theta \cos \phi \]
      b. Sketch the orbital corresponding to the wavefunction above.

8. Explain how a wave can have rotational energy and be a solution to the Schrödinger equation, but not possess angular momentum?

9. The energy of rigid rotor as a function of the rotational quantum number \( J \) is given by the following equation, where \( I \) is the moment of inertial.
      \[ \varepsilon_J = \frac{\hbar^2 J(J+1)}{2I} \]
      In addition, the degeneracy of each s, p, d, etc. quantum state is given by the following equation.
      \[ g_J = (2J+1) \]
      From statistical mechanics, the rotational partition function for rotation in one dimension is given by the following expression.
      \[ \q_{rot} = \sum_J g_J e^{\frac{-\varepsilon_J}{k_B T}} \]
      a. Use these collective relationships to prove that the internal energy from the total rotational degrees of freedom based on statistical mechanics is equal to \( 2Nk_B T \) at room temperature.
      b. What is the corresponding value for the rotational contribution to the heat capacity at constant volume?
      [Hint #1: The rotational constant for H₂⁺ is 29 cm⁻¹ compared to \( k_B T = 208 \) cm⁻¹ at room temperature. Hint #2: consider the substitution \( u = J(J+1) \) when evaluating the integral].
# Periodic Classification of the Elements

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* Lanthanides
† Actinides

(Numbers in parentheses are the mass numbers of the most stable isotopes.)