Department of Chemistry  
Cumulative Examinations  

February 8, 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 **FOUR** examinations.

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

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 BRWN 2124.
#1 (50 pts) Basics of photons and waves. Please answer the following questions:

a. Why does the sky look blue in a sunny day?

b. Two laser pens. One is green, the other is red. Which laser beam has higher energy per photon?

c. Of the following properties: wavelength, frequency, polarization, energy, which one describes the particle behavior of a photon?

d. What kind of wave is used computed tomography (CT)?

e. What kind of wave is used in magnetic resonance imaging (MRI)?

#2 (50 pts) The Lycurgus glass cup (British Museum, 4th Century AD) has a unique optical property: When illuminated from outside, it appears green. When illuminated with white light from inside, it glows red. It is known the cup contains gold nanoparticles in the glass. The extinction spectrum of a gold nanoparticle solution is shown below. Please answer the following questions.

![Image of Lycurgus glass cup]

- The very small amounts of gold nanoparticles have an extinction peak at about 520 nm.

- The graph shows the extinction coefficient (A_u) of 60 nm Au NPs as a function of wavelength (nm).

a. Why does the cup appears green when illuminated from outside? Why does the cup glows red when illuminated from inside?

b. When passing light through a solution, light attenuation (i.e. extinction) is contributed by two physical processes, name the two processes.

c. Sketch a device to measure the extinction of a gold nanoparticle solution.

d. Name two methods to measure the size of these gold nanoparticles.

e. Gold nanoparticles have been widely use for surface enhanced Raman scattering (SERS). Describe the principle of SERS.

#3. (50 pts.) Microscopy based on the signal from molecular vibration provides a way to visualize specific molecules without fluorophore labeling. Please answer the following questions.

a. FTIR has the sensitivity of detecting single monolayer of molecules. However, it is not suitable for imaging live cells or tissues? Why?

b. Raman scattering include Stokes and anti-Stokes scattering. Which one is spectrally overlapped with the one-photon fluorescence emission background?

c. What are the three vibrational mode of the CH₂ group? Which mode has the highest vibration frequency?

d. A student is using a laser at 800 nm to do Raman imaging. The Raman shift of the amide I band is 1650 cm⁻¹. What is the wavelength of the Raman scattering from the amide I band?

e. Raman microscopy has better spatial resolution than FT-IR microscopy. Why?
1. In the recently prepared Zintl phase $K_6ZnBi_5$ the structure shows infinite chains of $K$ cations capped by $[ZnBi_4]^3^-$. The $[ZnBi_4]^3^-$ was claimed as an all metal aromatic species analogous to the tetrazole $[HCN_4]^-$ and $Bi_5^-$. Show bonding in $HCN_4^-$ and $Bi_5^-$ and explain why they are aromatic. Contrast that with $[ZnBi_4]^3^-$. Finally assign oxidation states to the elements that make up the Zintl phase compound $K_6ZnBi_5$.

2. Peroxynitrite is made from the reaction of nitrous acid and hydrogen peroxide (see below). Peroxynitrite is short lived. It decomposes to nitrate. A signal in the UV-vis was observed for HOONO allowing its monitoring. A biexponential fit for the rise and disappearance of HOONO gave two first-order rate constants 3.8 s$^{-1}$ and 0.085 s$^{-1}$. How can you decipher which constant belongs to step one ($k_1$) and step two ($k_2$)? Show clearly your reasoning and what experiments you would carry out.

$$\text{HONO} + H_2O_2 \underset{k_1}{\overset{k_2}{\rightarrow}} \text{HOONO} \rightarrow H^+ + NO_3^-$$

3. A ruthenium carbene complex (see structure below) was recently published as a catalyst for water oxidation using $Ce^{4+}$ as the oxidant. Write a balance chemical equation for water oxidation with cerium. The kinetics for water oxidation were found to be second order overall, first order in $Ce^{4+}$ and first order in the ruthenium complex. In most Ru bipyridine catalysts, it is believed that a dinuclear Ru complex is responsible for water oxidation. In the case of the carbene complex here, the authors argued that a single Ru site is responsible for activity. What information is given here that supports that claim? What oxidation state is ruthenium in the complex below? Propose a mechanism for water oxidation with the Ru carbene complex.

![Ruthenium Carbene Complex](image)
Organic Cumulative Exam

February 2014

Theme: Natural Products Chemistry
(with focus on recent works in Org. Lett, JACS, and Angew. Chemie Int. Ed.)

1. (30 pts.) Hung-Wen Liu and coworkers have identified GDP-α-D-gluco-octose as a key intermediate in the biosynthesis of lincomycin (J. Am. Chem. Soc., 2014, 136 (3), pp 906–909). They were able to establish the following facts, presented in reverse order of the figure below:

- 2-Octulose-8-phosphate is made from ribose-5-phosphate and fructose-6-phosphate by a transaldolase (LmbR);
- Octose-1,8-bisphosphate (Oct-1,8-bisP) is generated from 2-octulose-8P by an isomerase (LmbN) and a phosphatase (LmbP);
- GDP-α-octose is made from Oct-1,8-bisP by a kinase and GTP transferase (LmbK, LmbO).

\[ \text{Ribose 5-phosphate} + \text{Fructose 6-phosphate} \xrightarrow{\text{LmbR}} \text{Octulose 8-phosphate} \xrightarrow{\text{LmbN}} \text{Octose 1,8-bisphosphate} \xrightarrow{\text{LmbP}} \text{GDP-octose} \]

a) (10 pts.) Draw 2-octulose-8P in extended (linear) form with the correct stereochemistry, guided by the starting material and product structures.

b) (20 pts.) LmbN and LmbP are essentially acid/base catalysts, only a lot more sophisticated. Based on that information, provide a working mechanism for the conversion of 2-octulose-8P into Oct-1,8-bisP (for simplicity, you can substitute the enzymes with "H^+" and/or "B.").

2. (30 pts.) Moobery, Cichewicz and coworkers used a "crowdsourcing" initiative to collect and screen soil samples submitted by volunteers (Angew. Chem. Int. Ed. 2014, 53(3), 804–809). As a result, they identified a novel fungal metabolite (maximiscin, 1) having strong activity against several cancer cell lines (mean GI_{50} < 0.4 μM). Maximiscin is classified as a hybrid metabolite, generated by the shikimate and polyketide synthesis pathways. Maximiscin was characterized by NMR and found to exist as two slowly interconverting conformational isomers (however, the chemical shifts of the P and M isomers are nearly overlapping).

a) (15 pts.) Use the NMR data provided for the P isomer to establish the correct structure, choosing from one of four structural isomers A–D (see next page), with a brief explanation.

b) (15 pts.) Draw a fragment of maximiscin indicating the positions of carbons 13 and 14, and protons 14a and 14b.
Possible structures for maximiscin (1)

Select NMR data for (P)-1 (500 MHz, DMSO-$d_6$)

<table>
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<th>No.</th>
<th>$\delta_H$ (J in Hz)</th>
<th>$\delta_C$</th>
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<td>13</td>
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<tr>
<td>14b</td>
<td>4.62, dd (10.3, 1.8)</td>
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<td>3.92, brs</td>
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<td>5'</td>
<td>3.96, dd (1.5, 6.2)</td>
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<td>6'</td>
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2D-ROESY of maximiscin
3. (40 pts.) Liang et al. discovered a novel polycyclic aromatic norditerpene, paoveitol (Org. Lett. 2014, 16(2), 424–427). The following spectroscopic data is provided:

- HR-ESIMS: [M+Na]$^+$ = 351.1203
- IR: notable (v. strong) peaks at 3426, 1630, 1179 cm$^{-1}$
- $^1$H, $^{13}$C NMR: see Table 1 for chemical shifts, peak areas ($^1$H), and select couplings/J values.
- 2D NMR: HSQC, HMBC, and ROESY spectra.

a) (20 pts.) Using the available information, propose a molecular formula, unsaturation number, and total number of rings (including aromatic ones) in paoveitol.

b) (5 pts.) Briefly explain why some of the $^{13}$C chemical shifts (C2, C5, C11, C14) are above 150 ppm.

c) (15 pts.) To the best of your ability, propose a structure for paoveitol. Partial credit will be awarded for substructures that are backed up by spectroscopic evidence.

<table>
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<th>Table 1: summary of NMR data for paoveitol</th>
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<td>19</td>
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</table>
HSQC spectrum of paevetol (1) (600/150 MHz, pyridine-d₅)

HMBC spectrum of paevetol (1) (600/150 MHz, pyridine-d₅)
ROESY spectrum of paeoveitol (1) (600 MHz, pyridine-$d_6$)
Physical Chemistry (Quantum Mechanics)

1. (10 pts) We have learned that simple models based on quantum mechanics can be applied to calculate different energies in molecules therefore to predict optical excitations corresponding to transitions between those energy levels. Match the model with the energy problem.

- Particle-in-a-box model
- Particle-in-a-ring model
- Harmonic oscillator model
- Particle-on-a-sphere model

- vibration transition in HF
- rotation transition in HF
- optical absorption wavelength in ethene
- optical absorption wavelength in benzene

2. (10 pts) What is the total probability of finding a hydrogen 1s electron \textit{within} a radius of 5\(a_0\)? Write down the integral for calculating this probability first, and then circle the correct answer. 
   A. Close to 0  
   B. Close to 0.5  
   C. Close to 1

3. (30 pts) A free electron confined in a 1-D box has ground state energy of 1.5\(\times\)10\(^{-18}\) J.
   a. What is the length of the box?
   b. Predict the ground state energy of a proton, rather than an electron, confined in the same 1-D box.
   c. What is the expectation value of the kinetic energy of the above electron?
   d. What is the expectation value of the potential energy of the above electron?
   e. What is the expectation value of the position of the above electron (assuming that the box extends from 0 to the box length)?
   f. Roughly estimate the \textit{uncertainty} in the momentum of the above electron?

4. (20 pts) What is the ground state term symbols of N?

5. (30 pts) Consider the following two chemical reactions:
   
   A. \(6 \text{ H} \leftrightarrow 3 \text{ H}_2\)
   
   B. \(6 \text{ H} \leftrightarrow 2 \text{ cyclic-H}_3\)

   a. Use the Hückel approximation to express the ground state energy of \(\text{H}_2\) in terms of the Hückel parameters \(\alpha\) and/or \(\beta\).
   
   b. Use the Hückel approximation to express the ground state energy of \(\text{H}_3\) (cyclic) in terms of the Hückel parameters \(\alpha\) and/or \(\beta\).

   c. Express the energy, \(\Delta E\), of reaction A in terms of the Hückel parameters \(\alpha\) and/or \(\beta\).
   
   d. Express the energy, \(\Delta E\), of reaction B in terms of the Hückel parameters \(\alpha\) and/or \(\beta\).
FUNDAMENTAL CONSTANTS AND CONVERSION FACTORS:

\[ R = 8.3 \text{ J/(K mol)} \]
\[ h = 6.6 \times 10^{-34} \text{ J s} \]
\[ m_e = 9.1 \times 10^{-31} \text{ kg (electron mass)} \]
\[ e = 1.6 \times 10^{-19} \text{ C (electron charge)} \]
\[ \varepsilon_0 = 8.854 \times 10^{-12} \text{ C}^2/(\text{J m}) \]
\[ N_A = 6.0 \times 10^{23} \text{ molecules/mol} \]
\[ 1 \text{ Å} = 100 \text{ pm} = 0.1 \text{ nm} = 10^{-10} \text{ m} \]

\[ k_B = R/N_A = 1.38 \times 10^{-23} \text{ J/K} \]
\[ \hbar = h/2\pi = 1.1 \times 10^{-34} \text{ J s} \]
\[ m_p = 1.7 \times 10^{-27} \text{ kg (proton mass)} \]
\[ c = 3.0 \times 10^8 \text{ m/s} \]
\[ a_0 = 0.529 \text{ Å} = 0.0529 \text{ nm} = 5.29 \times 10^{-11} \text{ m} \]
\[ 1 \text{ eV} = 1.6 \times 10^{-19} \text{ J} \]

EQUATIONS:

Operators: \( \hat{x} = x \cdot \hat{p} = \frac{\hbar}{i} \frac{\partial}{\partial x} \hat{K}_x = \frac{-\hbar^2}{2m} \frac{\partial^2}{\partial x^2} \hat{V}_x = V(x) \cdot \hat{\mathbf{H}} = \hat{K} + \hat{V} \)

For any operator \( \hat{A} \) and normalized wave function \( \Psi \):

\[ \langle \hat{A} \rangle = \int \Psi^* \hat{A} \Psi \text{d}\tau \]

Uncertainty principle:

\[ \sigma_x \sigma_p \geq \frac{\hbar}{2} \]

1-D Particle in a box: \( n = 1, 2, 3, 4, \ldots \)

\[ E_n = \frac{n^2 \hbar^2}{8ml^2} \]

\[ \Psi_n = \sqrt{\frac{2}{L}} \sin \left( \frac{n\pi}{L} x \right) \]

Particle on a ring: \( n = 0, \pm 1, \pm 2, \pm 3, \ldots \)

\[ E_n = \frac{n^2 \hbar^2}{2l} \quad I = \mu r^2 \]

Harmonic oscillator: \( n = 0, 1, 2, 3, \ldots \)

\[ E_n = h\nu \left( n + \frac{1}{2} \right) = \hbar \omega \left( n + \frac{1}{2} \right) \]

\[ \omega = 2\pi \nu = \sqrt{\frac{f}{\mu}} \quad \nu = \frac{c}{\lambda} = \frac{c}{2\pi} \]

Particle on a sphere: \( l = 0, 1, 2, 3, \ldots \)

\[ E_l = \frac{\hbar^2}{2l} (l+1) \quad I = \mu r^2 \]

Hydrogen atom: \( n = 1, 2, 3, \ldots \)

\[ E_n = -\frac{\hbar^2 \nu}{n^2} \quad R_H = 109,737 \text{ cm}^{-1} \quad \Psi_{1s} = \Psi_{100} = \frac{1}{\sqrt{\pi a_0^3}} e^{-r/a_0} \quad P_{1s}(r) \text{d}r = \left| \Psi_{1s}^2 \right| 4\pi r^2 \text{d}r \]

Term symbol: \( ^{2S+1}L_J \)

Huckel determinants:

\[
\begin{vmatrix}
\alpha - E & \beta \\
\beta & \alpha - E
\end{vmatrix}
\]

\[
\begin{vmatrix}
\alpha - E & \beta & 0 \\
\beta & \alpha - E & \beta \\
0 & \beta & \alpha - E
\end{vmatrix}
\]

Roots:

\[ \alpha \pm \beta \quad \alpha, \alpha \pm \sqrt{2}\beta \quad \alpha + 2\beta, \alpha - \beta, \alpha - \beta \]
QUESTION 1 (25 POINTS TOTAL) Basic Kinetics  The enzyme paininthease has recently been purified from the tortured brains of chemistry students. The reaction catalyzed by this enzyme is reversible and the reaction sequence for this enzyme is shown below:

\[
\begin{array}{c}
\text{E} + \text{S} \underset{k_1}{\overset{k_2}{\rightleftharpoons}} \text{ES} \underset{k_{-1}}{\overset{k_2}{\rightleftharpoons}} \text{E} + \text{P}
\end{array}
\]

Part A (15 Points)
Derive the Steady-State rate equation for the above reaction in the forward direction. Assume initial rate conditions (i.e. \([ P ]_{\text{time}=0} = 0\)). Show ALL of your work and the necessary assumptions and equations. Place a simplified form of the final equation in the box on the next page.

Part B. (10 Points).
If a small amount of product were present in the reaction mixture at time zero, and if \(k_2 \gg k_{-2}\), would your final equation need to be adjusted? If no, then explain why? If yes, then explain how?
**QUESTION 2 (30 POINTS TOTAL) Basic Enzyme Kinetic Mechanisms**

**PART 3A. (20 points)** Draw the Cleland diagrams for each of the four Bi Bi kinetic reaction mechanisms that we discussed in class.

Here's an example of a Cleland diagram for a Uni Uni reaction to illustrate the form to use:

![Cleland Diagram](image)

**Ordered Sequential:**

**Ping Pong:**

**Theorell-Chance:**

**Random Sequential:**
**PART 3B. (10 points)** In the table below are product inhibition patterns for an enzyme reaction.

<table>
<thead>
<tr>
<th>Varied Substrate</th>
<th>Product Inhibitor</th>
<th>Inhibition Plot</th>
</tr>
</thead>
<tbody>
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<td>A</td>
<td>P</td>
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<td>A</td>
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</tr>
<tr>
<td>B</td>
<td>Q</td>
<td>Non-competitive</td>
</tr>
</tbody>
</table>

Which of the following kinetic enzyme mechanisms (from the 4 listed) does it describe:

**Ordered Sequential, Ping Pong, Theorell-Chance, or Random Sequential?**

Explain your reasoning in terms of the effects of product inhibition on $V_{\text{max}}$ and $K_m$ of the reactions.
QUESTION 3 (25 POINTS TOTAL) Basic Enzyme Inhibition
The following questions (A-C) are all based on the different types of enzyme inhibition.

Part A. (10 Points)
Fill out the remainder of each the kinetic schemes below for each of the following types of enzyme inhibition. Make sure you indicate the proper kinetic constants in your schemes.

Uncompetitive (Pure)  
\[
E + S \underset{k_2}{\overset{K_s}{\rightleftharpoons}} ES \rightarrow EP
\]

Non-competitive (Pure)
\[
E + S \overset{K_s}{\rightleftharpoons} ES \rightarrow EP
\]

Part B. (10 Points)
Fill in three lines for each of the plots below at fixed [I], varying [A]. Be sure to include an ARROW indicating the direction of increasing [I].

Uncompetitive (Pure)
\[
\frac{1}{v} \quad \frac{1}{|A|}
\]

Non-competitive (Pure)
\[
\frac{1}{v} \quad \frac{1}{|A|}
\]

Part C. (5 Points)
One of the following equations describes Competitive Inhibition. Please CIRCLE the correct one. Remember that \( k_{cat} \) is directly proportional to \( V_{max} \).

\[
v = \frac{k_{cat}[S]}{K_m + [S](1 + [I]/K_i)}
\]

\[
v = \frac{k_{cat}[S]}{K_m(1 + [I]/K_i) + [S]}
\]

\[
v = \frac{k_{cat}[S]}{(K_m + [S])(1 + [I]/K_i)}
\]
QUESTION 4 (20 POINTS TOTAL) Enzyme Chemistry

Draw out the complete reaction mechanism for a cysteine protease enzyme which uses a catalytic triad (Cysteine, Histidine and Aspartic Acid) to catalyze the hydrolysis of a peptide bond. Please show the amino acid side chains, their correct protonation states and the involvement of water molecules and reaction intermediates (if any).
## Periodic Classification of the Elements

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<th>IIA</th>
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*Lanthanides

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†Actinides

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<td>254</td>
<td>253</td>
<td>256</td>
<td>257</td>
<td></td>
</tr>
</tbody>
</table>

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