Department of Chemistry
Cumulative Examinations
September 21, 2013

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, Pages 1-2
2) Biochemistry Cumulative Examination, Page 3
3) Inorganic Cumulative Examination, Pages 4-5
4) Organic Cumulative Examination, Pages 6-11
5) Physical Cumulative Examination, Pages 12-13

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.
Analytical cume, September 2013

1. Draw energy level diagrams to indicate the transitions in infrared absorbance and in Raman spectroscopy.

2. Which of the following is most suitable for wavelength selection in Raman spectroscopy?
   a. Monochromator
   b. Dichroic Filter
   c. Michelson interferometer
   d. Prism
   e. CCD camera

3. For which of these is one most likely to use photon counting?
   a. UV Absorbance spectroscopy
   b. FTIR
   c. Raman spectroscopy
   d. X-ray ciffraction
   e. Microwave spectroscopy

4. Rank these techniques in order of sensitivity, listing them in order from lowest to highest sample concentration needed. Assume that the substance being analyzed has a reasonable response for the frequency of interest.
   a. UV absorbance spectroscopy
   b. FTIR
   c. X-ray ciffraction
   d. Fluorescence
   e. $^{13}$C NMR

5. Write the equation for the relation between wavelength, frequency and the speed of light.

6. Substance A has molar absorptivities of 20,000 and 2,000 L/(mol·cm) at wavelengths 300 and 500 nm. Substance B has molar absorptivities of 2,000 and 20,000 L/(mol·cm) at wavelengths 300 and 500 nm. For a 1-cm cell of an unknown mixture of the two substances, the absorbances were measured to be 0.21 and 0.12 at 300 and 500 nm, respectively. Calculate the concentrations of substances A and B.

7. The technician was performing an analysis using UV absorbance spectroscopy, where he took the blank reading and then took the sample reading. The lamp drifted to a higher intensity
between the blank and the signal measurements. Would this make his sample appear to be higher or lower in concentration? Briefly explain.

8. Explain in one sentence why the method of standard addition is often used in atomic emission spectroscopy but less often in inductively coupled plasma spectroscopy.

9. The sunset appears orange because of which optical phenomenon?
   a. absorbance
   b. scattering
   c. reflection
   d. refraction
   e. interference

10. A rainbow is caused by which optical phenomenon?
   a. absorbance
   b. scattering
   c. reflection
   d. refraction
   e. interference
Cumulative Examination in Biochemistry 21 September, 2013
Carbohydrate structure, properties and function in living organisms

Instructions: Use diagrams and appropriate examples to answer the following questions.

1. Carbohydrates can be depicted in a wide range of different representations and isomeric forms. Discuss this statement, using appropriate diagrams of one particular example of a carbohydrate to illustrate your answer.

2. What effect does temperature have on the isomerization of dissolved sugars? Use representations to illustrate your answer.

3. Do chain or ring structural forms of monosaccharides predominate in cells? Use specific examples to explain why.

4. Consider the solubility and chemical properties of monosaccharides, and predict what types of covalent and non-covalent interactions could occur if there was a persistently higher than normal concentration of glucose in the blood stream of a diabetic. Illustrate your answer with specific examples of structures and reactions.

5. Cellulose is often used as a laxative. What properties of cellulose make it a good laxative and why?

6. In order to break up sucrose into elemental carbon, what kind of experiment would you set up? Can living organisms do the same? If so, why? If not, why not?

7. Cells use specific oligosaccharides to encode important information about intracellular targeting of proteins, cell-cell interactions and extracellular signals. Discuss this statement illustrating your answer with any ONE specific example.
Inorganic Cumulative Exam

Qualitative molecular orbital theory and triple bonds.

1. Write Lewis structures for two triply bonded and neutral diatomic molecules of the form \( A_2 \) or \( AB \). They should be well known, as opposed to obscure systems.

2. Do the same for two neutrals that involve a run of three main-group atoms \( ABC \) in which the most stable resonance structure has a triple bond between atoms \( A \) and \( B \). (They do not have to be triatomic; \( A \) and/or \( C \) and be bonded to other atoms.)

3. Same as question 2 except both systems must be anions.

4. Same as question 3 except both systems must be cations.

5. Dimolybdenum complexes of the type \( \text{Mo}_2\text{X}_6 \), where \( \text{X} \) is a halide or pseudohalide, entail a metal-metal triple bond. What is the oxidation state and electron count at molybdenum? If metal-metal bonding occurs along the \( z \)-axis, show the three orbital overlap patterns, and identify the atomic orbitals involved.

6. Let the long axis of the linear molecule \( \text{H}-\text{C} \equiv \text{C}-\text{C} \equiv \text{C}-\text{H} \) be along the \( z \) direction. Enumerate the \( \pi \) molecular orbitals of the system and qualitatively indicate relative energies, with the highest energy MO(s) on top of the diagram. For each MO define directions for the \( x \)- and \( y \)-axes, and indicate contributing atomic orbitals and their relative phases (signs). (Don’t worry about relative amplitudes.) Identify each MO as overall bonding, non-bonding, or antibonding, and explain your reasoning. Finally, account for any degeneracies.

7. The following are a series of contours for the \( \text{Ph} \equiv \text{C} \equiv \text{C} \equiv \text{C} \equiv \text{Ph} \) molecule, where \( \text{Ph} \) denotes a phenyl group. As drawn, the molecule is planar. Explain why the HOMO and HOMO-1 are not degenerate and rationalize the relative energies. Same for the LUMO and LUMO+1.
8. Elemental boron is a solid, but it is possible to investigate the B₂ molecule in the gas phase. In the ground electronic state, it has two unpaired electrons. Draw a simple MO scheme consistent with this finding.

9. In theory at least, binding two molecules of CO to B₂ induces spin pairing and stabilizes a linear molecule, often described by the following Lewis structure. What kind of bonds do the arrows denote? This system also has a manifold of π molecular orbitals. Where appropriate, assign formal charges to atoms in the Lewis structure. Finally, sketch contours of the most stable and least stable π molecular orbitals within the scheme; assign relative phases and amplitudes to the contributing atomic orbitals. Assume axes as defined in the figure.
1. Snyder and Smith (*J. Am. Chem. Soc.* 2013, 135, 12964-12967), during their synthesis of (+)-ScholarisineA, have performed the following reactions for the synthesis of lactam 8 and ester 7 as shown in the attached article.

![Chemical Reaction Diagram](image)

(a) Write down structures for intermediates A and B

(b) Is product B formed as a single isomer? Rationalize stereochemical outcome.

(c) What is IBX? Write the full structure of IBX?

(d) Provide a plausible mechanism for this IBX reaction (B to 8)

2. How would you carry out this following transformation? Provide reagents and reaction intermediates for conversion of 8 to 18.

![Chemical Reaction Diagram](image)
3. Snyder and Smith have carried out the following reactions for the synthesis of Boc-amino ketone 16 from ketone 15 in 59% chemical yield. Then converted 16 to enone 17.

(a) Show step-wise mechanism of transformation of 15 to 16.
(b) Explain why 16 was formed as a major isomer.
(c) Draw the structures of TMG and TEMPO.
(d) Show the mechanism of transformation of 16 to 17.

4. During the late-stage radical C-H arylation reaction of 7 with nBu3SnH and ACHN, a minor product 23 was obtained in 6% yield. Show how this product was formed and explain why this product was formed as a minor isomer. Draw the structure of ACHN.

5. Diels-Alder reaction of 11 and 12 provided products 13 and 14 in approximately 3:1 ratio and around 83% yields.

(a) Draw the structure 14 with appropriate stereochemistry.
(b) How would you convert 14 to 13 (partial conversion is fine).
A Concise Total Synthesis of (+)-Scholarisine A Empowered by a Unique C–H Arylation

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Supporting Information

ABSTRACT: The structurally unique akuammiline alkaloid (+)-scholarisine A was synthesized in 14 steps from a known enone (15 steps from commercial materials) through a route empowered by a unique C–H arylation reaction to forge its polycyclic core. Additional key steps include a pyrone Diels–Alder reaction and a radical cyclization/Keck allylation to fashion the core cage polycycle and one of the molecule’s quaternary centers, as well as the use of a carefully positioned pendant hydroxyl group to facilitate the chemoselective reduction of an extremely unreactive lactam in the presence of a readily reduced lactone.

Monoterpenoid indole alkaloids have a long and storied history in treating disease, elucidating core biochemical pathways, and advancing the power of synthetic chemistry through efforts to fashion and derivatize their structural intricacies. One recent addition to the family is the akuammiline alkaloid scholarisine A (1, Scheme 1), a dense, polycyclic compound isolated from Alstonia scholaris, a plant used in traditional Chinese medicine to treat various respiratory diseases.1 Its structure is quite unique within the class2 given that it possesses an indolenine fused to a strained carbocyclic cage, itself adorned with several tertiary and quaternary stereocenters as well as potentially labile imine and bridging lactone functional groups; its specific biological properties, however, remain unexplored. Given this profile, it is unsurprising that chemists have been drawn to its challenges and its potential. In 2012, the Smith group developed an elegant, enantioselective total synthesis of (+)-1 which proceeded in 20 steps from a known compound (25 steps from commercial materials).3 Key elements of their approach, which constitutes the only complete solution to this target, are shown in Scheme 1. In addition, Higuchi and co-workers recently reported model studies toward the spiroindolenine portion of 1 using an oxidative coupling to forge that domain.4 Herein, as part of a program interested in rapidly accessing unique polycyclic frameworks,5 we report a distinct analysis of the synthetic challenges posed by scholarisine A. That approach sought to develop a unique C–H arylation to attack the indolenine domain directly to a near fully functionalized core, itself prepared rapidly and efficiently through the use of Diels–Alder chemistry and a radical cyclization/capture cascade.

Our retrosynthetic analysis of 1 is delineated in the lower part of Scheme 1. Describing its logic in the forward sense, we were guided by the general notion that the target’s core

Scheme 1. Unique Structure of Scholarisine A (1), Past Synthetic Approaches, and a Distinct Retrosynthetic Analysis of the Target Molecule

Received: July 1, 2013
Published: August 21, 2013
carbocyclic substructure could potentially be fashioned rapidly if we could access a material such as 10 with an oxabicyclo[2.2.2]octane. The key event for its preparation was envisioned to be an endo- and diastereoselective pyrrole Diels–Alder reaction, setting the stage for a tandem 6-exo-trig radical cyclization/Keck allylation to then access 9. This cascade operation was expected to form the quaternary center with high diastereoselection via exo-face delivery of the allyl group to an intermediate α-carbonyl radical. From here, completion of the last ring of the caged core through epimerization of the amine-bearing stereocenter and lactamization would enable attempts to directly incorporate the indolene domain via imine formation (to form 7) followed by a novel, late-stage tertiary C–H arylation reaction. Although such processes are known in specific contexts to occur with activated, enolizable carbons, to the best of our knowledge no examples involving nonenolizable, and potentially labile, imines are known. Moreover, as an additional challenge, positional selectivity between two different tertiary centers (the starred carbons within 7) would be required, assuming that the initial imine stereochemistry would not be product-determining. However, if these variables could be addressed successfully, then (+)-scholarsines A (1) could result from a final chemoselective reduction of a hindered lactam in the presence of a fairly accessible lactone. To aid in that task, the functionality of the strategically positioned allyl side chain could potentially prove useful.

As shown in Scheme 2, our synthetic efforts began with a Diels–Alder reaction between dienophile 11 available in decagram quantities in three steps from N-Boc-D-Serine, see Supporting Information) and a slight excess (1.2 to 1.8 equiv) of methyl cinnamate (12) at 100 °C in toluene for 33 h. These conditions afforded the desired product (13) alongside a minor amount of its separable diastereomer 14 and another very minor component of unknown structure (3:1:0.1), in 83% combined yield. A priori, this cycloadition would seem to be unfavorable, given the electron-deficient nature of both components; as such, it is notable that it proceeds with such efficiency. Moreover, the protecting groups on the serine-derived dienophile had a significant effect on both the yield and diastereoselectivity of the reaction; for instance, the corresponding O-TBS-protected congener of 11 afforded Diels–Alder adducts in 47% yield and a dr of 1:7:1:0.1.

With 13 in hand, we next set about transforming its protected alcohol into an appropriate radical cyclization precursor. That task was accomplished using TFA in CHCl₃ at 0 °C to remove the acetone chemoselectively followed by bromination under Appel-type conditions to afford bromide 15 in 86% yield. With the stage now set for our key cascade, we were pleased to find, after a screen of several carbon-based radical traps, that following exposure of 15 to allyltributylstannane and Et₃B as a radical initiator under an air atmosphere 16 could be obtained in 59% yield as a single diastereomer possessing the desired stereochemistry at the C-20 quaternary center (as confirmed by X-ray analysis).

Having reached this critical staging area, we next envisaged deprotecting the nitrogen atom and effecting epimerization/lactamization to access 8 directly through treatment with the appropriate base. However, exploration of various conditions along these lines revealed that the deprotected α-amino ketone underwent facile dimerization and oxidation instead to give pyrazine 18. Fortunately, a solution presented itself during epimerization attempts on 16 using DBU as base when we noticed that adventitious oxygen could readily oxidize this compound to its corresponding unsaturated congener 17, presumably due to the ease of forming a captodative radical intermediate; an optimized process using TMG and TEMPO in THF under air gave enone 17 in 68% yield. This outcome presented the possibility of a deprotection/exo-face reduction/lactamization sequence. Gratifyingly, treatment of a CH₂Cl₂ solution of 17 with NaBH₄CN and TFA at 0 °C followed by heating in EtOAc afforded the desired caged compound in excellent yield (91%) with concomitant reduction of the ketone to the endo alcohol (structure not shown). Although the latter event was undesired, control experiments showed that ketone reduction was necessary to prevent pyrazine dimer formation. The constitution of the full cage structure was secured by extensive 2-D NMR studies as well as X-ray analysis of a crystalline derivative. Oxidation with IBX then completed the synthesis of ketone 8 in >85% yield.

We were now poised to attempt the challenging late-stage arylation reaction to append the indolene domain onto our
Scheme 3. Completion of the Total Synthesis of (+)-Scholarine A (1)\textsuperscript{44}

Reagents and conditions: (a) 2-iodoaniline (1.1 equiv), PPTS (0.09 equiv), PhMe, THF, 4 Å m.s., 90 °C, 18 h; (b) n-Bu$_3$SnH (1.2 equiv), ACHN (1.2 equiv), toluene, 110 °C, 4.5 h, 25%; (c) Driver’s reagent, 110 °C; (d) O$_2$, -78 °C, 18 h; (e) Raney Ni; (f) NaBH$_4$; (g) NIS or IDSI; (h) [5-exo-trig cyclization]; (i) [5,6-H atom transfer]; (j) [5-exo-trig cyclization]; (k) NIS, 23. Although this intermediate (structure and absolute configuration verified by X-ray) ultimately proved to be unproductive in its own right, its synthesis prompted us to explore the allyl group, or some group derived from it, as a means to achieve chemoselective reduction of the lactam through intramolecular assistance. Thus, ozonolytic cleavage of the alkene and reduction of the resulting aldehyde to amino alcohol provided a potential handle in the form of a primary alcohol. Pleasingly, treatment with Lawesson’s reagent at 110 °C effected thiation/cyclo dehydration to deliver thiocyclic 26. Without that handle, the lactam groups of 22, or its dialoytetrathyldiethany derivatives, did not undergo thionation under identical (and even more vigorous) conditions.\textsuperscript{24} Subsequent excision of the S atom within 26 using Raney Ni in THF at 23 °C for 1 h simultaneously formed the ethyl group and imine function of 1 in the form of 27. A final oxidation using PhIO at ambient temperature then gave (+)-scholarine A (1) in near-quantitative yield, the spectroscopic data of which matching those of the naturally-derived material in all respects.

In conclusion, we have completed an enantioselective synthesis of (+)-Scholarine A (1) in 14 steps from known enone (15 steps from commercial materials). This route compares favorably in terms of step count to the inaugural Smith synthesis\textsuperscript{3} of 1. Key discoveries include an efficient and diastereoselective pyrrole Diels–Alder reaction to rapidly form the appropriately functionalized [2.2.2]-bicyclic, a radical cyclization/Keck allylation to concurrently forge the [3.3.1]-bicycle and C-20 quaternary center, an indoline annihilation at a nonenolizable tertiary center via a novel late-stage radical C-H arylation, and the use of a pendant hydroxyl group to facilitate the chemoselective reduction of an extremely unreactive lactam. Current and future work is directed toward applying the developed strategy to related targets, exploring the scope of the C–H arylation step, and probing the biochemical potential of scholarine A itself.
While its 2,6-dimethylaniline congener does so even more readily (110 °C). We expected our 2-iodoaniline derived imine to isomerize at an intermediate rate: (a) Wurmb-Gerlich, D.; Vogtle, F.; Mannscher, A.; Staab, H. A. Liebigs Ann. Chem. 1967, 708, 36. In addition, any residual acid present would be expected to facilitate the isomerization process: (b) Jennings, W. B.; Al-Showman, S.; Tolley, M. S.; Boye, D. R. J. Chem. Soc., Perkin Trans. 2 1975, 1535.

At the planning stage, we recognized that if isomerization of the imine geometric isomers was competitive (e.g., thermally), then the initial E/Z ratio may not be important. Staab and co-workers have shown that the imine derived from acetone and aniline isomerizes rapidly on the NMR time scale at higher temperatures (E/Z-Me signal coalescence temperature = 126 °C) while its 2,6-dimethylaniline congener does so even more readily (110 °C). We expected our 2-iodoaniline derived imine to isomerize at an intermediate rate: (a) Wurmb-Gerlich, D.; Vogtle, F.; Mannscher, A.; Staab, H. A. Liebigs Ann. Chem. 1967, 708, 36. In addition, any residual acid present would be expected to facilitate the isomerization process: (b) Jennings, W. B.; Al-Showman, S.; Tolley, M. S.; Boye, D. R. J. Chem. Soc., Perkin Trans. 2 1975, 1535.


In the absence of TEMPO, the reaction proceeded in ca. 40% yield. We suspect TEMPO is a more effective radical trap than O2, leading to less decomposition/side reactions.

Although this step might be envisioned to be difficult, condensations of anilines and 2-aminantamone, a material that is similar in structure to 8, are known. For a representative example, see: Sanaki, T.; Eguchi, S.; Hirako, Y. Tetrahedron 1976, 32, 437.

This transformation was achieved through oxindole, dihydroxylation, and desulfurization with Raney Nickel.

Selected reductions attempted: T3O/H/SiB3H7, CP2ZrHCl, LiAlH4, Red-Al, AH2, E/HNMe3, H2/THF, BH3/SmEt2; reduction of corresponding methyl imidate (MeOBF2): LiAlH4, BH2/THF, AH2, NaBH4, NaBH4/HCl, Zn/AlOH, LiDBB, Ra-Ni. In all cases no reduction of the lactam (or its derivative) was observed.


(10) At the planning stage, we recognized that if isomerization of the imine geometric isomers was competitive (e.g., thermally), then the initial E/Z ratio may not be important. Staab and co-workers have shown that the imine derived from acetone and aniline isomerizes rapidly on the NMR time scale at higher temperatures (E/Z-Me signal coalescence temperature = 126 °C) while its 2,6-dimethylaniline congener does so even more readily (110 °C). We expected our 2-iodoaniline derived imine to isomerize at an intermediate rate: (a) Wurmb-Gerlich, D.; Vogtle, F.; Mannscher, A.; Staab, H. A. Liebigs Ann. Chem. 1967, 708, 36. In addition, any residual acid present would be expected to facilitate the isomerization process: (b) Jennings, W. B.; Al-Showman, S.; Tolley, M. S.; Boye, D. R. J. Chem. Soc., Perkin Trans. 2 1975, 1535.

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Subject: Fundamentals of spectroscopy

Physical Chemistry Cumex Exam  September 2013

\( h = 6.6 \times 10^{-34} \text{ J s} \)

\( m_e = 9.1 \times 10^{-31} \text{ kg} \) (electron mass)

\( m_p = 1.7 \times 10^{-27} \text{ kg} \) (proton mass)

\( c = 3.0 \times 10^8 \text{ m/s} \)

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

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

1) (50 points) Consider an electron which is in the ground state of a one-dimensional box that extends from \( x=-b/2 \) to \( x=+b/2 \).

a) (5 points) Sketch the ground state wave function of the above electron.

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|----------------------------------|
-\( b/2 \) \hspace{2cm} +\( b/2 \)
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b) (10 points) What is the functional form of the above properly normalized wavefunction?

\( \Psi(x) = \)

c) (5 points) What is expectation value of the position of the above electron?

In answering the remaining questions (d-f) you may make use of the fact that the expectation value of the kinetic energy of the above electron is \( 6 \times 10^{-20} \text{ J} \).

d) (10 points) What is the value of \( b \) (expressed as a number in nm units)?

e) (10 points) What is the uncertainty (standard deviation) of the velocity of the above electron (expressed as a number in m/s units units)?

f) (10 points) Predict the optical absorption wavelength of the above system (as a number with nm units).
2) (30 points) The following figure shows the fluorescence and absorption spectra of a given molecule dissolved in a liquid.

\[
\begin{array}{c}
\text{Ground} \\
\text{Excited} \\
\end{array}
\begin{array}{cccccc}
\text{v} = & & & & & \\
\text{v} = & & & & & \\
\end{array}
\]

Intensity

\[
\text{Wavelength (nm)}
\begin{array}{cccccccc}
300 & 350 & 400 & 450 & \\
\end{array}
\]

a) (10 points) Fill-in the boxes in the above figure to indicate which is the fluorescence and which is the absorption spectrum.

b) (20 points) Fill-in the blanks to assign the vibrational quantum numbers of ground and excited electronic states which give rise to the corresponding prominent vibronic peaks in the fluorescence and absorption spectra.

3) (30 points) Consider the following atoms, molecules, or ions: $\text{H}$, $\text{H}_2$, $\text{H}_2^+$, $\text{c-H}_3$, $\text{n-H}_3$. Note that $n$-$\text{H}_3$ is a linear triatomic with two identical bond lengths and $c$-$\text{H}_3$ is a cyclic triatomic with three identical bond lengths.

a) (5 points) Which of the above molecules would you expect to have one or more allowed vibrational infrared absorption transition(s)?

b) (5 points) Which of the above molecules would you expect to have one or more allowed vibrational Raman scattering transition(s)?

c) (5 points) Which of the above molecules would you expect to have one or more allowed rotational microwave absorption transition(s)?

d) (5 points) Which of the above molecules would you expect to have one or more allowed rotational Raman scattering transition(s)?
Periodic Classification of the Elements

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*Lanthanides*  
- Ce (140.12)  
- Pr (140.97)  
- Nd (144.24)  
- Sm (150.35)  
- Eu (151.96)  
- Gd (157.25)  
- Tb (158.924)  
- Dy (162.50)  
- Ho (164.930)  
- Er (167.26)  
- Tm (168.934)  
- Yb (173.04)  
- Lu (174.97)  

†Actinides  
- Th (232.038)  
- U (238.03)  
- Np (237)  
- Pu (242)  
- Am (243)  
- Cm (247)  
- Bk (249)  
- Cf (254)  
- Es (253)  
- Fm (256)  
- Md (256)  
- No (256)  
- Lw (257)  

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