Syllabus of Chem5530, Spring 2020

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Meeting Time/Place: MR 2:30 – 6:20 pm, Widtsoe 336 and 346

Lab Fee: $75 US Dollars

Course Project: Synthesis, Characterization and Biological Study of Amphiphilic Kanamycins

Work Schedule and Tasks:

1) Students will work in pair and conduct experiments at the course meeting time.
2) Detailed experimental procedures, required reading and quizzes can be found on Canvas.
3) Spring break: March 2nd – 6th.
4) Dates for lab clean-up: April 20th and/or April 23rd.
5) Final report due: April 27th.

<table>
<thead>
<tr>
<th>Week</th>
<th>Experiment or Task$^a$</th>
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<tbody>
<tr>
<td>1</td>
<td>Safety training, setting up experiment, watching videos and journal reading$^b$</td>
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<tr>
<td>2</td>
<td>Synthesis of amphiphilic kanamycins, KI07 and K47$^c$</td>
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<tr>
<td>3</td>
<td>Synthesis of amphiphilic kanamycins KI07 and K47</td>
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<tr>
<td>4</td>
<td>Synthesis of amphiphilic kanamycins KI07 and K47</td>
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<tr>
<td>5</td>
<td>Synthesis Quiz, Synthesis of amphiphilic kanamycins KI07 and K47</td>
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<td>6</td>
<td>Synthesis of fluorescent KI10 and K47$^c$</td>
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<td>7</td>
<td>Synthesis of fluorescent KI10 and K47$^c$</td>
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<tr>
<td>8</td>
<td>Synthesis of fluorescent KI10 and K47$^c$</td>
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<td>9</td>
<td>Spring break</td>
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<tr>
<td>10</td>
<td>Observe growing of fungi and bacteria, Antibacterial and antifungal evaluation of kanamycin, KI07 and K47</td>
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<tr>
<td>11</td>
<td>Bioassay quiz, Antibacterial and antifungal evaluation of kanamycin, KI07 and K47</td>
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<td>12</td>
<td>Imaging of cells treated with KI10 and K47</td>
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<tr>
<td>13</td>
<td>Fluorescence quiz, Imaging of cells treated with KI10 and K47</td>
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<tr>
<td>14</td>
<td>Synthesis of inorganic complex and X-ray crystallography$^d$</td>
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<tr>
<td>15</td>
<td>Quiz, Synthesis of inorganic complex and X-ray crystallography$^d$</td>
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<tr>
<td>16</td>
<td>Reserved for incomplete tasks and lab clean up</td>
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The detailed experimental procedures and assigned papers for reading are in separate files posted in “Module” of the corresponding week. The journal can be downloaded from Canvas. Students will need to complete the online quizzes following the journal reading. NMR characterization can be conducted when needed; this will be performed in Dr. Berreau’s laboratory.

On-line Resources for Lab Techniques:

(http://ion.chem.usu.edu/~tchang/Chem5530/Chem5530.htm)

Re crystallization (http://www.youtube.com/watch?v=aAa065hWyYg)

Flash Column Chromatography (http://www.youtube.com/watch?v=fF1gXUvyGb4)

TLC Technique (http://www.youtube.com/watch?v=EUn2skAAjHk)

Laboratory Safety Requirements for Chem5530

General Safety Rules:
Please follow the rules and requirements listed on the departmental website for teaching lab at:

http://www.chem.usu.edu/safety/SafetyNew-working

Pregnancy:
Students that are pregnant are required to meet with the lab instructor to obtain a list of all reagents that will be used in the course. The student should then meet with their physician to make a final decision whether or not to complete the lab course while they are pregnant.

General Learning Objectives for Chem5530

<table>
<thead>
<tr>
<th>General Learning Objective</th>
<th>Examples of Specific Objectives</th>
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| Gaining factual knowledge (terminology, classifications, methods, trends) | a. Use of TLC to monitor the progress of reactions.  
b. Know how to operate the common work-up procedures.  
c. Know how to operate the common purification procedures  
d. Use NMR for structural characterization. |
| Learning fundamental principles, generalizations, or theories | a. Apply the knowledge in organic chemistry on bench-top experiments  
b. Know how the reactions provide the desired products.  
c. Know the safety precaution for operating hazardous reagents or operations. |
| Learning to apply course material (to improve thinking, problem solving, and decisions) | a. Know the rationale for the synthetic approaches.  
b. Know the applications of the synthetic tasks. |
Specific Learning Objectives:

Know how to record the experimental data.

Know how to monitor the progress of organic reaction

Know how to carry out column chromatography purification.

Know how to design a proper workup procedure.

Know how to report the experimental result.

Know how to find the related references.

Know how to interpret the spectroscopic data to support the experimental results

Grade Breakdown:

1. Required Criteria for Lab Notebook (20%):

1. Informative. All of the essential information needs to be incorporated, for example the reaction scheme, physical properties of compounds and reagents, amounts of compound and reagents used, reference, actual procedure, TLC information, column chromatography information, and NMR characterization data.

2. Easy to follow by others. Clarity is important. All the information needs to be written clearly so others can repeat and reproduce the same results.

3. Associated with NMR data. A tracking or coding system is needed for locating the NMR spectra with the corresponding experiments.

Template for reporting NMR spectroscopic data:

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.13 (dd, $J = 9.6, 9.1$ Hz, 1H, H-5), 5.0 - 5.1 (m, 2H), 5.04 (dd, $J = 9.8, 9.6$ Hz, 1H, H-4'), 4.94 (dd, $J = 9.9, 9.9$ Hz, 1H, H-6), 4.55 (d, $J = 10.3$ Hz, 1H, NH), 4.40 (ddd, $J = 9.9, 4.3, 4.1$ Hz, 1H, H-5'), 4.00 (ddd, $J = 10.5, 10.4, 3.7$ Hz, 1H, H-2'), 3.69 (dd, $J = 9.7, 9.7$ Hz, 1H, H-4), 3.6 (m, 1H, H-1), 3.5 (m, 1H, H-3), 3.3 - 3.4 (m, 2H), 2.44 (ddd, $J = 13.4, 4.6, 4.5$ Hz, 1H, H-2eq), 2.07 (s, 3H), 2.04 (s, 3H), 2.01 (s, 3H), 2.00 (s, 3H), 1.65 (ddd, $J = 13.1, 12.7, 12.6$ Hz, 1H, H-2ax), 1.43 (s, 9H, C(O)(CH$_3$)$_3$); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.9, 169.8 (2C), 169.6, 155.3, 98.9, 80.3, 77.7, 74.2, 73.7, 71.3, 70.2, 69.4, 58.8, 57.9, 53.0, 51.2, 32.0, 28.3, 20.8 (2C), 20.7 (2C)
Example of organizing NMR spectra:

2. Final Report (50%):

1) Use the course project for the final report.

2) **Important**: Use *J. Med. Chem.* from ACS as the template.

3) Submit your report electronically as WORD document. Submit your Supporting Information as a PDF file.

4) Quality of the manuscript will be judged based on clarity, explanation (rationale of design, approaches, etc), description of the results, conclusion and reference citing.

5) The experimental data needs to be organized according to the publication format. Please refer to the sample template/example above.
6) Students are encouraged to ask for assistance from USU Science Writing Center in writing the final report.

3. Online Quizzes (30%):

Grade Scheme

<table>
<thead>
<tr>
<th>Grade</th>
<th>A</th>
<th>A-</th>
<th>B+</th>
<th>B</th>
<th>B-</th>
<th>C+</th>
<th>C</th>
<th>C-</th>
<th>D+</th>
<th>D</th>
<th>F</th>
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<tbody>
<tr>
<td>% Scores</td>
<td>≥90%</td>
<td>≥85%</td>
<td>≥80%</td>
<td>≥76%</td>
<td>≥68%</td>
<td>≥60%</td>
<td>≥56%</td>
<td>≥50%</td>
<td>&lt;50%</td>
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