LABORATORY SYLLABUS

**CHEMISTRY 3514: Organic Chemistry I**

**FALL 2016**

**PROFESSOR:** Dr. Jerry Fong (Physical & Health Sciences Building). Phone:#: 587-3692.

E-Mail: FONGJD (campus); JERRYF6023@AOL.COM (home)

**Course Website:** [**http://web.alfredstate.edu/fongjd/OrgChem1.htm**](http://web.alfredstate.edu/fongjd/OrgChem1.htm)

**REQUIRED TEXTS:** **“Experimental Organic Chemistry: A Small Scale Approach,”**  C. F. Wilcox , 2nd edition

**GRADING:** Synthesis Labs: 100 -150 points each (350 pts) Technique Labs: 25 points each. (150 pts)

Two nail exercise 50 pts; Practicum 50 pts. Lab quizzes worth 3-5 pts each will be administered at the start of several labs and are part of the overall lab point total.

Laboratory is 30% of the course grade. **Flunk the Lab; flunk the Course**.

**LAB LEARNING OUTCOMES:**

1) Successfully perform the basic organic lab operations of recrystallization, melting point determination,

solvent extraction, steam and simple distillation and both IR and GC characterization.

2) Independently and successfully carry out a multi-step synthesis and characterization of a target organic compound given

a standard organic reaction kit and a standard organic preparatory procedure written in a fashion similar to your

lecture text. See also:”Two nail “exercise.

**ATTENDANCE:**

No make-up labs are allowed unless by express consent of the instructor. An unexcused lab absence converts to a `0’ (zero) for that lab. Two unexcused lab absences converts to an F for the **entire** course. I do not accept late labs. Late labs earn zeros (0).

**LAB PHILOSOPHY:**

Students often view Organic Chem lab as a kind of cooking class where you show up, follow the printed directions and hope the final `cookies’ don’t burn, explode or inadvertently become bagels. This leads to the common notion that synthesis is about 95% luck and 5% dumb luck. If you leave this course holding that opinion you’ll have entirely missed the point.

Consistently successful, efficient high yield/purity synthesis happens when you simultaneously master the underlying chemical mechanism that leads to the product (e.g., you know the `theory’ ) while at the same time firmly grasping how the described preparative techniques, conditions and individual procedures act to produce the each step (e.g, know the `practice’).

Knowledge is power in organic synthesis.

For the record, synthesis also includes an element of serendipity (okay, dumb luck.) Essentially you are trying to get ~1023 cranky, back-sliding electrons to perform a specific set of specific set of hop-skip-and-jumps at your command. Hence, variability in synthetic outcome is natural. However, chance favors the prepared mind. Good laboratory workers adjust and respond to what they see based on a firm knowledge of what to expect. They also exude a combination of preparation, deft organization, verbal acuity, independence of action and creative focus that-like good writing- can be recognized but only vaguely explicated. Bad laboratory workers exude the opposite traits. That too, is easily recognized and is reflected in crap yields and abysmally written reports.

**LAB REPORTS:**

Two kinds of laboratories comprise the curriculum: **Technique Labs** and **Synthesis Labs.** You will usually turn in your primary lab notebook for a **Technique Lab** at the end of lab except when products need drying time. For **Synthesis labs**, both your primary notebook and **a formal, typewritten `theory** for **Synthesis Labs 1** and **2** are required. For the **final Grignard synthesis** the primary notebook is not turned in, just the product itself along with characterizations for 25 pts. A 75 pt score for yield and purity will be assigned and a brief oral during the final labs for each student will be administered worth 50 pts. Grading rubrics for each lab will be made available.

As for writing up the **Technique Labs,** follow the example supplied by the instructor on how to write up a lab. I like the style captured therein. Mimic it if you can when. First person, singular is fine with me.

**REQUIRED LABORATORY EXPERIMENTS: CHEM 3514 FALL 2016**

PART 1: TECHNIQUE LABS (weeks 1-8) ~25 POINT/LAB

# /Week of **Lab Laboratory Topic Due in lab Read Before Lab Do in Lab**

**#1: 8/29** 1 Check-in; Safety;  *week 2* pp 3-9 ***Lab #1 Handout***

Handbook Lore *CRC look-it-up*

**#2: 9/5** 2 Melting Points & *9/11* *week 3* pp 84-5;87-97 ***Purification of*** Recrystallization ***of impure Acetanilide***

experiment C, p. 100

using method described in B p 98-9

**Measures: mp, color, habit**

**# 3: 9/12** 3 Boiling Points, *week 4*  pp 43-53;59-64 ***Distillation of pure ethanol***

Fractional Distillation; Experiment A, p. 66

Refractive index **Measures**: **bp, density, nD**

***Distillation of 1:1 ethanol/water***

Experiment B, pp 66-7

**Measures: bp, density, nD**

**#4: 9/19** 4 Steam Distillation *week 5* pp 77-8; 80-81 ***Separation of naphthalene &***

*Salicylic acid* ***by steam distillation***

Experiment B p 82

**Measures:% yield, mp**

**#5: 9/26** 5 Solvent Extractions *Wednesday* pp 104-106; 110-115 ***Separation of Benzoic acid***

week 6  ***and acetanilide by extraction*** (post break) Experiment B p.1171

**Measures: % yield, mp, color, habit**

1You will make up starting solution of 2 g benzoic acid and 2 g acetanilide/25 ml.. Instructor will provide 1.5 **M** KOH.

**#6 October Mini-break: No labs**

**#7: 10/10** 7 Instrumental Methods in ***Wed 10/8*** pp 234-244; 250-252 (IR)***Lab #6 Handout***:

Organic Chemistry: (post break) pp 122-123;(GC) ***Identification of an***

IR, GC pp 128-30;137-142 (GC) ***Organic Mixture via GC***

***and IR***

.eWe use a Spectrum 1 FTIR with ATR head and HP 6890 GC (50 m HP-1 capillary column +FID detector)

**Measures: annotated GC, IR**

**#8 : 10/17** \* **PRACTICUM (50 points)**

**Using just your lab notebook, you will randomly select one of the technique experiments above above**

**And carry out an analysis for the desired measures.**

**END OF `TECHNIQUE’ LABORATORY EXERCISES**

(150 Lab points total for Technique Labs + 50 pts for practicum)

\* **Thinking and writing like** **A synthetic chemist (50 pts): Handout: “The Two Nail Puzzle”**

**`Lab’ Writeup** **Due in lecture Wednesday 10/26**

PART 2: SYNTHESIS LABS (weeks 9-15) 100 -150 POINTS PER LAB

**#/Week of Laboratory Topic Due Date Read Before Lab Do in Lab**

**#9: 10/24** Synthesis of an Alkyl  **note book**  pp 309-312 P***reparation of n-Butyl Bromide***

Halide (100 pts) ***in lab week 10***  Experiment A: pp 312-31`4

**Theory 11/6**(Friday) **Measures: IR, % yield, nD, GC**

*notes:*

* *this one represents `synthesis boot camp’*
* *Be prepared for a long siege. It often runs overtime if you aren’t efficient\*
* *I expect at least 20 % yield of bona fide product or you will lose at least 30 points.*

***#10 10/31 Make-up lab if you blow boot camp***

**#11: 11/7** Synthesis of an Alkene **notebook**  pp 327-330 ***Preparation of Cyclohexene***

by Dehydration of an ***in lab week* *12 from Cyclohexanol***

Alcohol (100 pts)**Theory 11/20 (Friday)***Experiment A: p. 331*

**Measures: % yield, IR,GC, nD,**

**Baeyer and Br2 tests (show instructor)**

*notes:*

* *Yields vary from 20-80% here ! Separation technique is important*
* *I expect a minimum of 35% yield of bona fide product or you will lose at least 30 pts*

**#12: 11/14** Multi-step Synthesis by ***12/11***  pp 334-337 ***Preparation and Characterization***

of an Alkene to a ***of 2-Methyl-hexene from***

larger alcohol (150 pts) ***n-Butyl bromide and acetone***

Experiments B,C pp 337-339

**#14: 11/28** Multi step synthesis (cont) Preparation of final alkene: purification of crude hexanols

and dehydration to alkenes

Experiment D pp. 339-340.

**#15: 12/5** Multi step synthesis (cont) \*Characterization of final product

**Measures: IR, % yield**

**(show instructor yield + show**

**him Baeyer and Br2 tests)**

Experiment D, p 339-340

*notes:*

* *This one is synthesis bootcamp part II and can be dangerous.*
* *Read the Wilcox preparative procedure thoroughly.*
* *Student performance (e.g. yield) varies widely.*
* *Restarting is not uncommon.*
* *You can also carry out characterizations the week of 11/28 if you are done early. (You’ll need about 1 hour to do the three characterization tests).*

**#15: 12/5**  Check out (350 lab points total for Syntheses)