

# **PHARMACEUTICAL SCIENCE LABORATORY**

**(16219-310)**

## **LABORATORY MANUAL**

**Spring 2007**

**Department of Pharmaceutical Sciences**

College of Pharmacy, Nursing and Allied Health Sciences

School of Pharmacy

**Howard University**

**Washington, DC 20059**

*Pharmaceutical Science Laboratory 16219-310*

ii

**College of Pharmacy, Nursing and Allied Health Sciences**

**Howard University**

### **Course Plan**

## **PHARMACEUTICAL SCIENCE LABORATORY**

**Course Number:** 16219-310; Spring Semester 2006

**Coordinator:** Dr. LaVerne L. Brown (email: [ll\\_brown@howard.edu](mailto:ll_brown@howard.edu))

**Faculty:** E.O. Akala, Ph.D., M.J. Habib, Ph.D., G.J. Kapadia, Ph.D.,

**Credits:** Three (1-Hour Pre-Lab Lecture & pre-lab quiz +One 3-Hour

Laboratory & post lab assignment +1 post lab discussion and/or formal

report)

**Clock Hours per Week:** Pre-Lab conference- Tuesday 12:30-2:00 p.m.

Laboratory – 2:00-5:30 p.m. (on assigned day)

**Prerequisites:** None

**Required Textbooks:** Laboratory Manual

**Recommended Textbooks:** None

**Date Effective:** January 4, 2007

**COURSE DESCRIPTION:**

The course deals with the study, application and analysis of physico-chemical principles

governing the stability of pharmaceutical dosage systems.

**TERMINAL OBJECTIVES:**

At the end of the course, the student shall have acquired competency in the:

- (a) analysis of dosage systems and their components;
- (b) determination of the stability of dosage systems and their components;
- (c) mechanisms of stability of dosage systems and their components; and
- (d) the application of physico-chemical principles in the determination of stability of dosage systems and their components to provide hands-on training in the appreciation of the rational selection of the appropriate medication.

The student shall demonstrate this competency by the ability to:

1. Perform appropriate experiments to assess the physico-chemical properties in the determination of the stability of the dosage systems.
2. Provide a written summary of the experimental data.

*Pharmaceutical Science Laboratory* 16219-310

iii

3. Provide the answers to the study questions related to the experiment through a literature search.

4. Show comprehensive knowledge of the terminal objectives through quizzes and examinations.

#### COLLEGE-WIDE COMPETENCIES

Upon completion of laboratories, reading assignments, and other course-related activities, the student should be able to meet the following specific college-wide competencies:

1. Retrieve, evaluate and interpret health literature efficiently and accurately for pharmaceutical care, **research** and education.
5. Select, evaluate and recommend appropriate drug product(s) and/or **dosage form(s)** which are most useful for a disease state in an individual patient on the basis of biopharmaceutic, economic, **quality-control** and **assurance** as well as other relevant information.
6. Counsel and advise patients on the proper use, safeguard and handling of prescription and non-prescription (OTC) drug products, and home health care products.

#### RELATIONSHIP OF COURSE COMPETENCIES TO NAPLEX COMPETENCY STATEMENTS

The relationship of this course to NAPLEX competency statements should provide the student with those course-related activities that describe to the evolving role of the pharmacy profession toward patient drug therapy. These statements should provide the following competencies.

## **Area 1: Assure Safe and Effective Pharmacotherapy and Optimize Therapeutic**

### **Outcomes (Approx. 54% of Test)**

**1.2.0** Identify, evaluate, and communicate to the patient or health-care provider, the

appropriateness of the patient's specific pharmacotherapeutic agents, dosing regimens, dosage forms, routes of administration, and delivery systems.

**1.2.5** Identify physicochemical properties of drug substances that affect their solubility, pharmacodynamic and pharmacokinetic properties, pharmacologic actions, and stability.

**1.2.6** Interpret and apply pharmacodynamic and pharmacokinetic principles to calculate and determine appropriate drug dosing regimens.

*Pharmaceutical Science Laboratory* 16219-310

iv

## **Area 2: Assure Safe and Accurate Preparation and Dispensing of Medications**

### **(Approximately 35% of Test)**

**2.1.0** Perform calculations required to compound, dispense, and administer medication

**2.1.1** Calculate the quantity of medication to be compounded or dispensed; reduce and enlarge formulation quantities and calculate the quantity of ingredients needed to compound the proper amount of the preparation.

**2.1.4** Calculate or convert drug concentrations, ratio strengths, and/ or extent of ionization.

**2.2.0** Select and dispense medications in a manner that promotes safe and effective

use.

**2.2.4** Interpret and apply pharmacokinetic parameters and quality assurance data to determine equivalence among manufactured drug products, and identify products for which documented evidence of inequivalence exists.

**2.3.0** Prepare and compound extemporaneous preparations and sterile products

**2.3.1** Identify and describe techniques and procedures related to drug preparation, compounding, and quality assurance.

**2.3.2** Identify and use equipment necessary to prepare and extemporaneously compound medications.

**2.3.3** Identify the important physicochemical properties of a preparation's active and inactive ingredients; describe the mechanism of, and the characteristic evidence of incompatibility or degradation; and identify methods for achieving stabilization of the preparation.

## BEHAVIORAL OBJECTIVES

Upon completion of the lectures, conferences, reading assignments, and other course-related activities, the student should be able to satisfactorily meeting the following behavioral objectives;

1. Using the analytical balance, weigh a solid and liquid within an accuracy of + 0.1 mg.
2. Titrate, weigh, standardize and identify the correct end-points for unknown solutions in the determination of the % ascorbic acid in vitamin C tablets.
3. Attain knowledge in the use of medicinal chemistry techniques for the

synthesis, filtration, and % yield determination of acetylsalicylic acid (aspirin).

*Pharmaceutical Science Laboratory* 16219-310

v

4. Attain knowledge in the theory and practical uses of chromatography techniques.
5. Attain knowledge in the techniques used for alkaloid extractions.
6. Demonstrate an understanding of solubility and purification processes in the isolation of flavanoids.
7. Demonstrate the proper technique in leveling the prescription balance.
8. Using the prescription balance, determine the sensitivity requirement of the balance both empty and with a ten (10) gram load.
9. Demonstrate the proper technique in weighing using the prescription balance.
10. Determine the percent deviation in measurements derived from: (a) a prescription bottle; (b) a cylindrical graduate; (c) a teaspoon; and (d) a syringe.
11. Understand Tate's Law in the assessment of various liquids.
12. Demonstrate the proper technique in calibrating a prescription dropper.
13. Prepare and explain the molecular forces involved in Strong Iodine solution U.S.P. XXI.
14. Using a standard aqueous benzoic acid: sodium benzoate ion solution, determine the effect of pH on the aqueous solution.
15. Using a standard hydroalcoholic benzoic acid: sodium benzoate ion solution, determine the effect of pH on the hydroalcoholic solution.

16. Using a standard sodium benzoate solution, at a constant pH, determine the effect of alcohol on the concentration of the solutes.
17. Prepare isotonic solutions by the following methods: (a) Sprowl's method; (b) freezing point depression method; and (c) sodium chloride equivalent method.
18. Identify the compounds which release histamine from the mast cells and provide their mechanism of action.
19. With the assistance of the laboratory instructor, conduct a potentiometric titration of the  $pK_2$  of monosodium phosphate and determine the buffer capacity of the  $H_2PO_4$   
-1: $HPO_4$   
-2 buffer pair.
20. Compare the partition coefficient when the "single" versus "multiple extraction" methods are employed.
21. Determine the rate of aspirin hydrolysis at various temperatures.
22. Determine the surface tension of various solvents.

*Pharmaceutical Science Laboratory* 16219-310

vi

23. Determine the effect of non-ionic surface active agents on the solubility of pharmaceutical preparations that would normally be insoluble in the aqueous solution.
24. Determine the viscosity of various solvents.
25. Compare the compatibility of: (a) non-ionic polymer; (b) an anionic polymer;

and (c) amphoteric polymer in pharmaceutical systems.

26. Demonstrate the differences between deflocculated particles.

27. Demonstrate the effect of "wetting agents" on the processes observed in objective 36.

28. Determine the "Bancroft Rule" in the formation of: (a) a w/o; and (b) an o/w emulsion.

29. Determine the HLB system in the formation of: (a) a w/o; and (b) an o/w emulsion.

*Pharmaceutical Science Laboratory* 16219-310

vii

Pharmaceutical Science Laboratory 16219-310

LECTURE/LABORATORY SCHEDULE (SPRING 2006)

**Instructor MONTH DAY DATE ASSIGNMENT**

Brown January Tu 9 Laboratory Safety Requirements

Pre-Lab Experiment #1

T, W, Th (9-11) Check In

Experiment #1:

Metrology #1 - The Analytical Balance

Brown Tu 16 Pre-Lab Experiment #2

T, W, Th (16-18) Experiment #2:

Determination of Ascorbic Acid in Vitamin C

Tablets

Brown Tu 23 Pre-Lab Experiment #3

T, W, Th (23-25) Experiment #3:

Synthesis of Acetylsalicylic Acid (aspirin)

Kapadia Tu 30 Pre-Lab Experiment #4

Jan/Feb Lab sessions (30,31,1) Experiment #4:

Chromatography Theory

Kapadia February Tu 6 Pre-Lab Experiment #5

Lab sessions (6-8) Experiment #5:

Extraction of Alkaloids Theory

Kapadia Tu 13 Pre-Lab Experiment #6

Lab sessions (13-15) Experiment #6:

Isolation of Flavanoids

**Brown/**

**Kapadia**

**Tu 20 Examination #1**

**Experiments covered: #1- 6**

*Pharmaceutical Science Laboratory* 16219-310

viii

Pharmaceutical Science Laboratory 16219-310

LECTURE/LABORATORY SCHEDULE (Continued)

**Instructor MONTH DAY DATE ASSIGNMENT**

Habib Tu 27 Pre-Lab Experiment #7

Feb, March T, W, Th (27, 28, 1) Experiment #7:

Metrology #2 – The Prescription Balance

Habib March Tu 6 Pre-Lab Experiment #8

T, W, Th (6-8) Experiment #8 - Pharmaceutical Solutions

Habib Tu 13 Pre-Lab Experiment #9

T, W, Th (13-15) Experiment #9:

pK<sub>a</sub> Determination;

Buffer and Buffer Capacity;

Extraction phenomena (demonstration)

Tu 20 NO CLASSES SPRING RECESS

NO CLASSES SPRING RECESS

Akala Tu 27 Pre-Lab Experiment #10

T, W, Th (27-29)

Experiment #10:

Chemical Kinetics

Akala April Tu 3 Pre-Lab Experiment #11

T, W, Th (3-5) Experiment #11:

Surface Tension;

Rheology

Akala Tu 10 Pre-Lab Experiment #12

T, W, Th (10-12) Experiment #12:

Suspensions and Emulsions

**Habib/**

**Akala**

**Tu 17 Examination #2**

## **Experiments covered: #7-12**

Tu 24 Check Out – ALL SECTIONS

Course/Instructor Evaluations

*Pharmaceutical Science Laboratory* 16219-310

ix

## **GRADING SYSTEM**

The final grade computation for the course will be based on the following:

Examination #1 25%

Examination #2 25%

Quizzes 25%

Post Lab assignments\*/

Laboratory reports 25%

\* Laboratory reports submitted after the due date will be penalized 10% of the earned

grade. Additional penalties of up to 45% of the post laboratory assignments may accrue

for the following deficiencies:

1. Poor preparation = 10%
2. Failure to maintain a clean and neat work area = 5%
3. Breakage of Glassware and Equipment = 5%
4. Improper Handling of Weight Set = 5%
5. Lack of Accuracy in Weighing and Measuring = 5%
6. Improper Handling of the Balances = 5%
7. Lack of Accuracy and Completeness of Labeling = 5%

8. Poor Product Quality = 5%

The numerical equivalent of letter grades shall be:

90 – 100% = A

86 – 90% = B+

80 – 85% = B

76 – 79% = C+

70 – 75% = C

Below 70% = F

Incomplete = I

*Pharmaceutical Science Laboratory* 16219-310

x

## EXAMINATION/GRADING POLICIES

1. The examination schedule shall be distributed to the class at the beginning of the semester. The class (through the President), may request changing any or all of the scheduled semester examinations to the coordinator because of conflict with other courses only within the first ten (10) working days of the semester. The coordinator shall attempt to accommodate this request while taking into account the need to include a reasonable amount of course material in each examination. After the final revision, no change in the examination schedule shall be permitted except under unforeseen circumstances authorized by the course coordinator.

2. All students shall have the responsibility for presenting themselves regularly and promptly to take the scheduled examinations of the course. To ensure fairness in the conduct of examination, no tardy student shall be allowed into the examination room after any student has completed the examination and has left the room.

3. A student who fails to appear and take the examination on its scheduled date and time shall earn a grade of "zero".

4. An absence from the final examination shall be considered "excused" if it occurs because of any of the following circumstances:

(a) hospitalization of the student due to illness, birth of a child and/or accident;

(b) death in the immediate family (e.g., spouse, children, parents, guardians, brothers and/or sisters) supported by appropriate documentation; or

(c) summons to appear before the court.

5. In cases other than those listed in 4 (above), the absentee student shall submit a written petition to the course coordinator explaining the circumstances of missing the final examination and shall attach documents supporting his/her claim. The coordinator of the course shall then take the following actions:

(a) Consider the absence as "excused" and allow the student to take a make up examination with a MAXIMUM SCORE stipulation. In this case, the student shall earn the maximum score if his/her actual score is higher than this score and shall earn his/her actual score if it is lower than this score

(b) Consider the absence "unexcused". In this case the student shall be given a score of "zero" in the examination.

*Pharmaceutical Science Laboratory* 16219-310

xi

## PROCTORING EXAMINATIONS

All examinations shall be proctored by a minimum of two (2) faculty or staff members to

prevent and discourage cheating. If cheating is suspected, all proctors will act as

witnesses in accordance with procedures set forth in the Howard University Academic

Code of Conduct.

## PROFESSIONAL CONDUCT

Proper professional conduct in the laboratory is required. Unprofessional conduct will not be tolerated. It is disrespectful, disruptive, and may also cause injury to your distracted neighbors. Loud and/or profane language is unacceptable in a professional setting.

Therefore, students engaged in this conduct will be asked to leave the laboratory and will receive a grade of "zero" for the entire laboratory exercise. Subsequently, further participation in the laboratory will be pending the result of a written petition by the student to the course coordinator and the laboratory instructor.

## DRESS

PROFESSIONAL attire is required for class. Students inappropriately dressed will be asked to leave the laboratory. Students are required to wear **clean**, long white laboratory coats at all times in the laboratory.

## CHECK OUT

All students are required to report to class at the scheduled date and time to complete the check out process. An Incomplete grade (I/F) will be given to any student who does not complete the following:

- (1) all equipment washed and dried;
- (2) Equipment check list completed;
- (3) Instructor/course evaluation completed; and
- (4) Sign attendance sheet(s) to assure compliance with (2) and (3) (above).

## AMERICANS WITH DISABILITIES STATEMENT

Howard University is committed to providing an educational environment that is accessible to all students. In accordance with this policy, students in need of accommodations due to a disability should contact the Office of the Dean for Special Student Services for verification and determination of reasonable accommodations as soon as possible after admission to the University, or at the beginning of each semester. The Dean of Special Student Services, Dr. Barbara Williams, can be reached at (202) 238-2320.

## LABORATORY SAFETY RULES

The laboratory is a potentially dangerous place. However, the risk can be minimized if you are aware of the hazards and you follow safe laboratory practices.

Your safety depends on your knowledge of possible dangers and your adherence to certain safety rules. The major hazards of the laboratory are the following:

A. Some compounds are toxic, bio-hazardous, carcinogenic or corrosive.

Exposure to these chemicals can occur by inhalation or skin contact as well as by ingestion.

B. Some compounds or solvents are flammable. Their vapors can saturate the laboratory atmosphere or linger on benches or in sinks.

C. Broken glass may cause cuts and exposure to hot glass or metal may cause severe burns.

D. Electrocutation may occur when electrical instruments are mishandled.

E. You must provide for your individual use in the laboratory the following:

Safety gloves

Paper or Cloth Towels

Soap or Detergent

Weight Set

Long, White Laboratory Coat

Glass marking pen

## **The Following Safety Rules Must Be Followed At All Times**

1. No student is allowed to work in the laboratory unless a Faculty Member, a Laboratory Technician or a Teaching Assistant is present.

2. Before doing any laboratory work, carefully read these safety rules and other safety information in your laboratory exercises. Know the hazards associated with the materials you are using. Carefully read the label before using a chemical. Review the Material Safety Data Sheet (MSDS) for any special handling information.

Use hazardous chemicals only as directed and for their intended purpose.

Never smell or taste a hazardous chemical.

Inspect equipment or apparatus for damage before adding a hazardous chemical or beginning a hazardous procedure. Do not use damaged equipment or glassware.

Label all secondary containers appropriately

3. The laboratory should be treated as a professional pharmacy. Keep it neat and clean.

Wipe up spilled chemicals immediately.

Keep balances and work areas clean.

Do not throw powdered drugs, paper, greasy or waxy materials, or an insoluble waste material into the sink. Wrap these items in a paper towel and discard them in the waste container provided for this purpose.

Never pour unused solutions or other liquids back into the original container, pour them into the waste disposal container in the hood.

4. Conduct yourself as a pharmacist. Dress neatly.

Men are required to wear dress clothes.

Both men and women must wear long, white laboratory coats at all times while in the laboratory.

Caps, hats or other outdoor headgear are not to be worn in the laboratory.

5. Sandals or other open-type footwear may not be worn because of the hazard from broken glass and spilled, caustic chemicals.

6. Smoking, eating or drinking in the laboratory is absolutely forbidden.

7. Eye protection must be worn at ALL times in the laboratory.
8. Long hair must be secured.
9. Avoid contact with chemicals on your skin. Wash immediately if chemicals come in contact with your skin or clothing.
10. Know the location of fire extinguishers, safety showers, eyewash stations, and first aid kits, and know how to use them.
11. Report any accident, however minor, to the instructor at once.

*Pharmaceutical Science Laboratory* 16219-310

xiv

### **Rules and Procedures for Conduct in the Laboratory**

1. An apparatus list will be supplied to you to check your apparatus against this list by first placing all of the equipment on the counter top and checking it off as you replace each item. Obtain the assistance of your instructor or laboratory technician in the event that you cannot identify an item or if an item appears to be missing.
2. After checking your equipment, prepare a list of missing items. Present the list to the laboratory technician. When your locker is complete and in order, request an instructor to check it out.
3. Prescription balances will be assigned to you. These are delicate and costly instruments. It is your responsibility to keep them clean. Observe all of the precautions for their care and use. Do not attempt to repair or adjust any balance issued to you. Ask for assistance when necessary.
4. When stock containers are removed from the spaces reserved for them, be sure to return them to their proper place as soon as you are through. Solid material removed from such containers must only be placed upon weighing paper or watch glass that has been labeled previously with the name of the material.
5. In order to avoid contamination of reagents, never return excess material to a stock bottle unless advised to do so by an instructor.
6. Work independently unless otherwise directed.
7. Avoid unnecessary noise and unnecessary movement in the laboratory.

8. Laboratory periods are to begin at the specified time and your presence at the prelaboratory discussion is required. Tardiness will not be tolerated. You are considered late when you arrive more than 10 minutes after the beginning of the prelaboratory discussion. You will be assessed a grade of zero for the exercise and will not be eligible for a makeup laboratory.

9. There will be no working in the laboratory other than at the scheduled times unless under the supervision of the instructor in charge.

10. Headsets are absolutely forbidden.

11. Each student is responsible for maintaining the laboratory in a clean, neat, and orderly condition.

12. It is important that students familiarize themselves with each exercise prior to the laboratory period and do any background reading and preparation deemed necessary to facilitate the smooth conduct of the laboratory. The pre-laboratory lecture is intended to clarify the day's assignment and to provide additional information relevant to the exercise.

*Pharmaceutical Science Laboratory 16219-310 xv*

**13. Pre-lab quizzes will be administered at the end of each pre- lab lecture.**

**Students are expected to earn a passing grade on the pre-lab quiz before attempting the assigned lab exercise. Failure to attend a pre-lab lecture shall result in a grade of zero on the pre-lab quiz.**

14. Completed post laboratory assignments must be submitted for grading at the prescribed times unless otherwise noted.

a) Tuesday Laboratory---No later than 3:00pm on the following Thursday.

b) Wednesday Laboratory---No later than 3:00pm on the following Friday.

c) Thursday Laboratory---No later than 3:00pm on the following Monday.

If the Monday is a holiday, then the post laboratory assignments shall be due at 9:00am on Tuesday. Post laboratory assignments and/ or formal reports submitted after the scheduled due date will be penalized 10% of the earned grade.

**15. Laboratory Attendance:** All students are expected to attend the laboratory sessions regularly and promptly in his/her assigned lab section. If absent from a laboratory session, the missed laboratory work must be made up, provided the

absence is excused (see below), the student's laboratory grade for the semester will be assessed an 8% penalty for each missed laboratory experiment.

**16. Excused Absences from laboratory sessions:** Absences from examinations and laboratory sessions shall only be under unavoidable circumstances such as:

- a) Hospitalization of the student due to illness, birth of a child and an accident;
- b) Death in the immediate family (spouse, children, parents, guardians, brothers, and sisters) supported by the appropriate documentation; and
- c) Summons to appear before the court for causes other than traffic adjudication.

Under other extraordinary circumstance, the absentee shall make written appeal to the coordinator of the course. If granted an excused absence, the missed laboratory assignment(s) shall be made up without penalty to the student.