18 PHARMACEUTICAL PREPARATIONS

18.1 Objective

18.1.1 To familiarize the trainee with the analytical procedures for pharmaceutical preparations

18.2 Modes of Instruction

18.2.1 Self-directed study through study questions and practical exercises

18.2.2 Presentations and demonstrations

18.3 References

18.3.1 *Physician's Desk Reference.* Montvale, N. J.: Medical Economics, various editions.

18.3.2 *Identadrug*, hardcopy series and website subscription.

18.3.3 *Drug Identification Bible.* Grand Junction, CO: Amera-Chem, Inc., various editions including CD versions.

18.3.4 DEA Logo Index, various editions.

18.3.5 Epocrates, website subscription.

18.3.6 Pillbox – National Library of Medicine website.

18.3.7 Poison Control Center

18.3.8 DEA *Microgram Bulletin*, various editions.

18.3.9 Moffat, A. C., editor. *Clarke's Isolation and Identification of Drugs.* London: The Pharmaceutical Press, 1986.

18.3.10 Clarke, E. G. C. *Isolation and Identification of Drugs, Volumes 1 and 2*. London: The Pharmaceutical Press, 1978.

18.3.11 Budavari, Susan, editor. *The Merck Index, Eleventh Edition.* Rahway, N. J.: Merck & Co., Inc., 1989.

18.3.12 DFS Controlled Substances Procedures Manual, Pharmaceutical Identifiers Section.

18.4 Assignments

Study questions and practical exercises

18.5 Study Questions

18.5.1 List the active ingredients of the following preparations:

- Desyrel
- Oxycontin
- Adderall
- Demerol
- Preludin
- Ritalin
- Viagra

- Keflex
- Percodan
- Paxil
- Dalmane
- Librax
- Valium
- Fiorinal
- Wellbutrin
- Zoloft
- Vicodin
- Suboxone
- Xanax

18.5.2 List four possible references for tablet logo identification.

18.5.3 What information should be recorded in the case notes to ensure proper documentation of visual examination?

18.5.4 What are the analysis and reporting requirements for tablets and capsules in Schedules II – VI?

18.5.5 What steps should be taken if the results of an analysis are inconsistent with the manufacturer's specification with regard to content?

18.5.6 How does the analysis of an injectable dosage form differ if tampering is suspected?

18.5.7 What are the most accurate sources for determining the schedule of a drug?

18.6 Practical Exercises

18.6.1 Obtain 5-10 unknown preparations from the TC. Perform the visual examination with references from two sources. Include the schedule of each component.

18.6.2 Obtain a sample of an injectable dosage form from the TC. Following the procedure in the procedures manual, analyze the item for possible tampering. Determine the relative concentration of the drug present by using semi-quantitative TLC or UV.

18.7 Mode of Evaluation

18.7.1 Written examination

19 EXTRACTIONS

19.1 Objective

19.1.1 To familiarize the trainee with the sample extraction methodology

19.2 Modes of Instruction

19.2.1 Self-directed study through reading assignments and study questions.

19.2.2 Practical exercises

19.3 References

19.3.1 Moffat, A. C., editor. *Clarke's Isolation and Identification of Drugs*. London: The Pharmaceutical Press, 1986.

19.3.2 Clarke, E. G. C., *Isolation and Identification of Drugs,* London: The Pharmaceutical Press, 1972, Vol. 1, 2.

19.3.3 Higuchi, T. et al. "Ion Pair Extraction of Pharmaceutical Amines" *Analytical Chemistry,* Vol. 39, 1967, p. 974.

19.3.4 Watson, D. G. *Pharmaceutical Analysis* New York: Churchill Livingstone, 1999, pp. 17-47.

19.3.5 Virginia Department of Forensic Science Controlled Substances Procedures Manual, ¶¶ 6, 23, 24, 25 and 29.

19.4 Assignments

19.4.1 Completion of required reading assignments

19.4.2 Study questions and practical exercises

19.5 Study Questions

19.5.1 What is a matrix?

19.5.2 What is the difference between recrystallization and precipitation?

19.5.3 Define the following with respect to filtration:

- Supernatant
- Filtrate
- Porosity
- Retentivity
- Speed

19.5.4 Describe how a mixed solvent recrystallization is performed. How is a single solvent recrystallization performed?

19.5.5 Why is it necessary to have at least two test tubes in a centrifuge?

19.5.6 Define the following:

- Unsaturated solutions
- Saturated solutions

- Supersaturated solutions
- Reflux
- Azeotrope

19.5.7 What is the difference between evaporation and sublimation?

19.5.8 What problems may be encountered if ether evaporates to dryness?

19.5.9 What is a dry extraction?

19.5.10 What effect does temperature have on a drug extraction?

19.5.11 Describe how a series of smaller volume immiscible solvent extractions is more efficient than a single extraction using the same total volume of organic solvent, using the concept of 'partition coefficient' in your description.

19.5.12 How can water be removed from organic solvents?

19.5.13 What is an emulsion? How can they be prevented and what can be done when one occurs?

19.5.14 What does "salting out" mean?

19.5.15 What does pH stand for? pK_a?

19.5.16 Describe how a pH controlled extraction works explaining equilibriums that are set up between two immiscible solvents.

19.5.17 Describe an extraction scheme that would recover most non-volatile compounds from an unknown.

19.5.18 What types of functional groups cause a compound to be acidic? Basic?

19.5.19 What does amphoteric mean?

19.5.20 Tell whether the drugs listed in Appendix A are acidic, basic, or neutral.

19.5.21 How is morphine best extracted from powder form?

19.5.22 How does hydrogen bonding come into play in liquid-liquid extractions?

19.5.23 What is ion-pairing? Diagram how it works using equilibrium considerations.

19.5.24 What types of factors should be considered in selecting solvents to use in extractions.

19.5.25 What separation advantages does chromatography have over extraction procedures? Disadvantages?

19.5.26 Describe how a soxhlet extractor works.

19.5.27 Describe the acetic acid extraction of psilocybin mushrooms emphasizing areas of concern.

19.5.28 What solvent should be used to extract salvinorin A from *Salvia divinorum* and why?

19.5.29 What is an extraction blank/procedural blank and when should it be used?

19.5.30 What extraction can be used to isolate water insoluble drugs from PEG solutions?

19.6 Practical Exercises

19.6.1 Obtain a Fiorinal with codeine capsule from the TC. Diagram a suitable extraction scheme using acid/base extractions. Perform these extractions to isolate each component into an organic solvent. Confirm each component utilizing either GC/MS or FTIR.

19.6.2 Obtain a sample of mushrooms from the TC. If none are available, use a standard of psilocybin. Perform the acetic acid extraction as outlined in the procedures manual. Confirm the presence of psilocyn.

19.6.3 Obtain a sample of a cocaine mixture from the TC or designee. Perform a dry extraction, and confirm using FTIR.

19.6.4 Obtain a sample of a food product containing THC. Perform the extraction outlined in the procedures manual. Confirm the presence of THC.

19.6.5 When available, perform the suggested Khat extraction on a sample of plant material. Confirm the presence of Cathinone and Cathine.

19.7 Mode of Evaluation

19.7.1 Written examination

20 COURTROOM TESTIMONY

20.1 Objectives

20.1.1 To familiarize the trainee with the functions of a courtroom criminal proceeding

20.1.2 To have the trainee prepare a current curriculum vitae and convey *voir dire* questioning during testimony

20.1.3 To familiarize the trainee with proper methods of presenting expert testimony during direct examination

20.1.4 To familiarize the trainee with the proper methods of defending analytical results during cross-examination

20.2 Modes of Instruction

20.2.1 Self-directed study through reading assignments and study questions

20.2.2 Observation of expert testimony

20.2.3 Practical exercises (mini-mock trials)

20.3 References

20.3.1 Kuzmack, Nicholas T., J.D., M.A. "Legal Aspects of Forensic Science", in Saferstein, Richard, Ph.D., editor. *Forensic Science Handbook.* Englewood Cliffs, N.J.: Prentice Hall, 1982, pp. 1-27.

20.3.2 Shellow, James M. "The Expert Witness in Narcotics Cases", in *Contemporary Drug Problems – A Law Quarterly*, Spring 1973, pp 81-104.

20.3.3 Travnikoff, Basil, Jr. and Kvick, Robert J. *How to Examine a Chemist in Drug Abuse Cases, First Edition*, 1971.

20.3.4 Bailey, F. L. and Rothblatt, H. B., *Handling Narcotic and Drug Cases*, Rochester, NY: The Lawyers Cooperative Publishing Co., 1972.

20.3.5 *Code of Virginia* (§ 19.2-187.1) and (§ 19.2-187).

20.4 Assignments

20.4.1 Completion of required reading assignments (20.3.1 – 20.3.3)

20.4.2 Completion of curriculum vitae

20.4.3 Study questions and mock trials

20.5 Study Questions

20.5.1 Discuss the role of the following during a trial:

- Expert witness
- Judge
- Prosecutor
- Defendant
- Defense counsel
- Jury

20.5.2 Define the following:

- *Voir dire*
- Direct examination
- Cross examination
- Redirect
- Chain of custody

20.5.3 Write a curriculum vitae (CV) in the format provided by the TC which includes educational background and work experience.

20.5.4 Describe a typical process from arrest to arraignment.

20.5.5 Describe a typical courtroom proceeding for a trial dealing with an individual accused of possession of a controlled substance, from the time the trial begins until final verdict by the jury. Be sure to include the order in which witnesses are called, arguments by trial counsel, and introduction of physical evidence.

20.5.6 How would you describe the characteristics of an effective expert witness? Likewise, what are some of the factors which make a poor expert witness?

20.5.7 Describe the ASCLD/LAB accreditation process and the benefits of being an accredited laboratory.

20.5.8 Briefly discuss the importance the *Melendez-Diaz v. Massachusetts* decision played in forensic science testimony.

20.6 Practical Exercises

20.6.1 Conduct several mock trials in conjunction with the TC or designee which deal with the following aspects of testimony separately:

- *Voir dire*
- Chain of custody
- Drug analysis

20.6.2 Conduct several mock trials which will encompass all aspects of a potential trial setting. Be sure to include role players to serve as judges, attorneys, and jurors.

20.6.3 Observe examiners testify whenever possible.

20.6.4 Verbally answer the following possible direct examination questions to the TC or designee:

- What is your name?
- What is your occupation? For whom do you work?
- How long have you been so employed?
- What are your duties in this occupation?
- What education and training do you possess that qualifies you to perform your duties?
- What specific courses have you taken that are directly related to drug analysis?
- How are these courses related? For example, what did you learn in your general chemistry course that aids you in the analysis of drugs?
- Do you consider yourself an expert in the analysis of drugs?
- What is the definition of an expert witness?
- Is the university/college you graduated from accredited, and if so, by whom?
- Who conducted your training?
- What are his/ her/ their qualifications?
- What literature do you read relating to your job?
- How many analyses have you done on suspected drugs (or controlled substances)?

- Do you belong to a recognized society attesting to your qualifications as a drug chemist?
- Have you written any articles or published materials dealing with your work?

20.7 Mode of Evaluation

20.7.1 Passage of final mock trial

21 SPECIAL TECHNIQUES AND ANALYSES

21.1 Objective

21.1.1 To familiarize the trainee with the theory and application of additional instrumental techniques which are either used infrequently or not currently available at the DFS laboratory, such as UV and NMR.

21.2 Modes of Instruction

21.2.1 Self-directed study through reading assignments

21.2.2 Study questions

21.3 References

21.3.1 Moffat, A. C., editor. *Clarke's Isolation and Identification of Drugs*. London: The Pharmaceutical Press, 1986, pp. 201-220; 221-236; and 264-275.

21.3.2 *Basic Training Program for Forensic Chemists*, U.S. Department of Justice, Drug Enforcement Administration, Office of Science and Technology, pp. 5-1 through 5-15; 5-49 through 5-59; 5-73 through 5-85.

21.3.3 DFS Controlled Substances Procedures Manual, Ultraviolet Spectroscopy Section.

21.3.4 Cooper, James. *Spectroscopic Techniques for Organic Chemists*. New York: John Wiley & Sons, 1980, pp. 53-135; 227-248.

21.3.5 Mills, Terry, III and Roberson, J. Conrad. *Instrumental Data for Drug Analysis, Second Edition.* New York: Elsevier, 1987.

21.3.6 Mills, Terry, III and Roberson, J. Conrad. *Instrumental Data for Drug Analysis, Third Edition*. New York: CRC Press, 2006.

21.3.7 Silverstein, R. M. et al. *Spectrometric Identification of Organic Compounds.* New York: John Wiley & Sons, 1991.

21.4 Assignments

21.4.1 Completion of required reading assignments (21.3.1, 21.3.3)

21.4.2 Study questions

21.5 Study Questions

21.5.1 Discuss briefly the theory of UV and NMR.

21.5.2 Draw the basic components of UV and NMR instrumentation and discuss the functions of each.

21.5.3 Describe the type of data provided by UV and NMR and illustrate with examples.

21.5.4 Which of the above techniques would you consider to be confirmatory and/or non-confirmatory? Why?

21.5.5 Obtain two UV spectra from the TC and provide as much interpretation as possible.

21.5.6 Obtain an NMR spectrum from the TC and provide as much interpretation as possible.

21.6 Mode of Evaluation

21.6.1 Completion of study questions

22 ADDITIONAL TRAINING

22.1 Review of Other Disciplines

22.1.1 During the course of the Training Program the trainee should spend a small amount of time with the other disciplines (latent prints, forensic biology, etc.) located within the Department. This will allow the trainee to understand how evidence is maintained for multi-sectional analysis as well as understanding the general capabilities of the other sections. These visits will be coordinated by the TC.

22.2 DEA Forensic Chemist Seminar

22.2.1 Upon completion of training, the trainee will be strongly encouraged to attend the DEA Forensic Chemists Seminar. Information of current schedules is found in *Microgram Bulletin* or by contacting the DEA Special Testing and Research Laboratory.

22.3 Technical/Administrative Review Training

22.3.1 The following documents shall be read and discussed with the TC or designee:

- Quality Manual - Section 17 Monitoring Results
- Technical Review Form
- ISO/IEC 17025:2005 – Section 4.13 Control of Records
- ASCLD/LAB-*International* Supplemental Requirements for the Accreditation of Forensic Science Testing Laboratories (2011) - Section 4.13 Control of Records

22.3.2 Practical Exercises

22.3.2.1 The trainee should document the review of at least twenty case files using the appropriate Technical Review Form. Case files should be generated by multiple examiners, if possible. The potential findings of the reviews shall be discussed with the TC. Technical Review forms generated in this capacity shall be marked as Training and retained in their Training File. The case files shall be technically reviewed by an authorized examiner pursuant to QM 17 prior to release.

23 CLANDESTINE LABORATORIES

23.1 Objectives

23.1.1 To familiarize the trainee with syntheses routinely used in clandestine laboratories.

23.1.2 The completion of this section is not required in order for the trainee to become a qualified examiner.

23.2 Modes of Instruction

23.2.1 Study questions and practical exercises

23.3 References

23.3.1 DFS Controlled Substances Procedures Manual, Clandestine Laboratories Section.

23.3.2 Weaver, K. and Yeung, E. *An Analyst's Guide to the Investigation of Clandestine Laboratories, 3rd edition.* Health Protection Branch, Ontario Region Health Canada, 1995.

23.3.3 *Clandestine Lab Basic Guide*, presented at the 12th Annual Clandestine Laboratory Investigating Chemists Training Seminar, 2002.

23.3.4 Ely, Roger, *et al*. *A Review of the Syntheses and Analyses of Phenyl-2-propanone, Amphetamine, and Methamphetamine.* Clandestine Laboratory Investigating Chemists, 1995.

23.3.5 Clandestine Laboratory Investigating Chemists monographs.

23.3.6 Strike. *Total Synthesis II*, San Antonio, TX: Panda Ink, 1999.

23.3.7 Uncle Fester. *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture.* Port Townsend, WA: Loompanics Unlimited. 1998.

23.3.8 Code of Virginia § 18.2-248.

23.3.9 Christian, Donnell R., Jr. *Forensic Investigation of Clandestine Laboratories*. CRC Press. 2004.

23.3.10 http://www.dfs.virginia.gov/laboratory-forensic-services/controlled-substances/meth-labs/

23.3.11 Angelos, S.A. et al. "The Identification of Unreacted Precursors, Impurities, and By-Products in Clandestinely Produced Phencyclidine Preparations", *Journal of Forensic Sciences*, 35(6), 1990, pp.1297-1302.

23.3.12 Skinner, H. F. "Methamphetamine Synthesis Via Hydroiodic Acid/Red Phosphorous Reduction of Ephedrine", *Forensic Science International*, Vol. 48, 1990, pp. 123-134 (found in CLIC: A Review of Syntheses and Analyses of Phenyl-2-Propanone, Amphetamine, and Methamphetamine. Vol. 1).

23.3.13 Person, E.C., Knops, L.A., Northrop, D.M., "One-Pot Methamphetamine Manufacture", *Journal of the Clandestine Laboratory Investigating Chemists Association*, Vol. 14, Number 2, April 2004, p. 14-15.

23.3.14 Ely, R. A. and McGrath, D. C., "Lithium-Ammonia Reduction of Ephedrine to Methamphetamine: An Unusual Clandestine Synthesis," *Journal of Forensic Sciences*, JFSCA, Vol. 35, No. 3, May 1990, pp 720-723.

23.3.15 Bremer, N. and Woolery, R. J., "The Yield of Methamphetamine, unreacted Precursor and Birch By-Product with the Lithium-Ammonia Reduction Method as Employed in clandestine Laboratories", *MAAFS Newsletter*, Fall 1999, pp 8-16

23.4 Assignments

23.4.1 Study questions and practical exercises

23.5 Study Questions

23.5.1 Describe the difference between a controlled precursor and List 1 or 2 chemicals.

23.5.2 Define the following terms:

- Precursor
- Byproduct
- Catalyst
- Limiting reagent

23.5.3 Explain how an analyst should sample a liquid with three layers.

23.5.4 Discuss the importance of working closely with prosecutors and officers to decide the amount of analyses necessary.

23.5.5 Review the Department's Clan Lab submission guidelines. Discuss the following:

- What items should/should not be submitted?
- What items will be analyzed?

- What weight thresholds are important in the manufacturing charges in Virginia?
- How should submitted items be packaged?

23.5.6 List chemicals and starting materials which would indicate the various syntheses of PCP and methamphetamine. What byproducts would be expected from these syntheses and why?

23.5.7 Discuss the types of analysis that may be necessary when the charge listed is Code of Virginia § 18.2-248 (J). Which compounds listed would be analyzed in the Controlled Substances section and which would be transferred to the Trace Evidence section?

23.6 Practical Exercise (optional due to availability of reagents/starting materials and laboratory safety)

23.6.1 Perform a synthesis procedure that is commonly encountered in clandestine laboratories (either methamphetamine or PCP is recommended).

23.6.1.1 Take samples during the reaction process to monitor the progress.

23.6.1.2 Determine the yield of the reaction.

23.6.1.3 Attempt to identify all compounds in the product mixture.

23.7 Mode of Evaluation

23.7.1 Completion of the study questions

24 FORENSIC LAB SPECIALISTS

24.1 Introduction

Forensic Lab Specialists (FLS) provide important support to Forensic Scientists in the laboratory. Typically FLS perform duties including, but not limited to the following:

24.1.1 Prepare solutions, reagents and standards

24.1.2 Participate in the quality assurance/quality control program

24.1.3 Maintain inventory of expendable supplies, reagents and materials

24.1.4 Perform routine maintenance of instrumentation and equipment

24.1.5 Perform general housekeeping duties (e.g., cleaning glassware, removing sharps waste)

24.1.6 Transfer sealed evidence

24.1.7 Under close supervision, perform routine procedures in the analysis of casework.

24.2 Training Outline

24.2.1 Instruction will be provided to the FLS by directed study, demonstration by the trainer and observation of the trainee. All tasks are performed under the direction of the trainer until the training segment is completed.

24.2.2 Reference information for the topics below can be found in other sections of this manual, the Controlled Substances Procedures Manual (CSPM) and other Department manuals. The specific locations are noted by section numbers in parentheses by the topic.

24.2.3 Orientation

24.2.3.1 Introduction to the local facility, staff and how the FLS fits into the Department of Forensic Science (DFS).

24.2.3.2 Description of the FLS position and clarification of duties.

24.2.3.3 Coverage of the following:

- Quality Manual
- Controlled Substances Procedures Manual with emphasis on the Quality Assurance Section
- Controlled Substances Training Manual
- Regional Operating Procedures
- Safety Manual, to include Bloodborne Pathogen and Chemical Hygiene training
- Organizational Chart of DFS

24.2.3.4 Introduction of the technical capabilities of all the DFS laboratories and how it fits into the Virginia law enforcement system.

24.2.3.5 Introduction to the LIMS system.

Appendix A – List of Known Drugs

- Cocaine HCl
- Cocaine Base
- Lidocaine
- Benzocaine
- Procaine
- Tetracaine
- Amphetamine
- Methamphetamine
- Methylphenidate
- Phentermine
- Ephedrine
- Pseudoephedrine
- Caffeine
- Theophylline
- Secobarbital
- Butalbital
- Acetaminophen
- Ibuprofen
- Aspirin
- Salicylamide
- Dextromethorphan
- Fentanyl
- Testosterone Propionate
- Nandrolone
- Substituted Cathinones
- Quinine
- Diphenhydramine
- Morphine
- Heroin
- Oxycodone
- Hydromorphone
- Codeine
- Hydrocodone
- Methadone
- Meperidine
- Guaifenesin
- Alprazolam
- Diazepam
- Phencyclidine
- LSD
- LAMPA
- Mescaline
- Psilocin
- Psilocybin
- Bufotenine
- 3,4-MDA
- 3,4-MDMA
- 4-Bromo-2,5-dimethoxyphenethylamine
- Ketamine
- Benzylpiperazine
- Amoxicillin
- Dimethyl Sulfone
- Cannabimimetic Agents