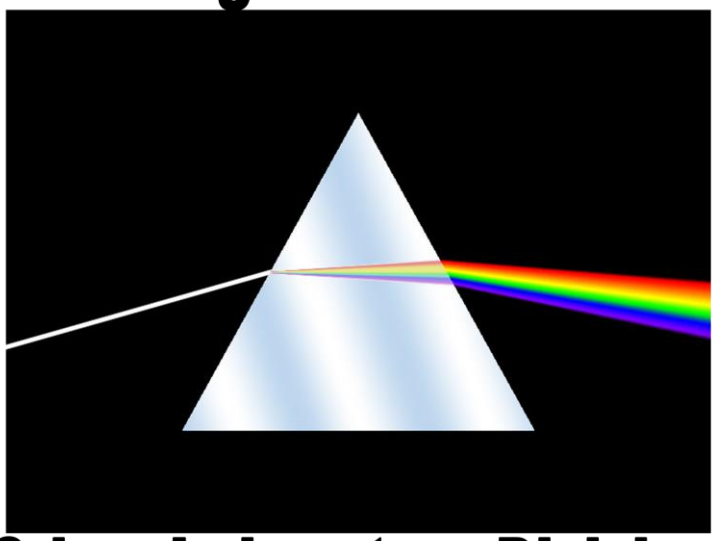
Washington State Patrol



Crime Laboratory Division

WASHINGTON STATE PATROL
CRIME LABORATORY DIVISION
Clandestine Laboratory Analysis Training Maual

November 2017

1. MANUAL OVERVIEW

1.1. Purpose and Scope

1.1.1. This manual contains an outline for training and/or assessing a forensic scientist in the area of clandestine laboratory analysis. Each scientist will have a unique training program depending on the individual's strengths and weaknesses, previous background, the needs of the laboratory, and available personnel to provide the training. The sequence in which the various sections are presented should not necessarily be considered as a mandatory order of training.

1.1.2. This manual endeavors to promote and maintain consistency and quality among forensic scientists performing clandestine laboratory analyses across the Crime Laboratory Division. Certain inherent aspects of chemical analysis prohibit the establishment of a rigid set of standard procedures to cover every case. Sufficient latitude should be given to allow for independent thought and individual freedom in selecting alternative courses of action. Upon completion of this training program, the trainee will be thoroughly familiar with the options available to perform an examination of most types of evidence that may be received.

1.2. Organization of the Training Manual

The training manual consists of several study segments, each covering different aspects of chemical analysis for clandestine laboratory evidence. Each study segment is comprised of five parts:

1.2.1. The *Objectives* summarize the purpose of each training segment.
1.2.2. The *Required Readings* section lists the reference material that must be read to successfully complete the study segments. The reading assignments are cumulative; comprehension of prior readings may be required to successfully complete study/discussion questions and exercises of subsequent study segments.
1.2.3. The *Suggested Readings* section lists reference material that may be read to assist in comprehension and successfully completing the study segments. It may not be necessary or practical to read every reference listed. The trainee will work with the trainer for specifics.
1.2.4. The *Study/Discussion Exercises* have a number of purposes:
- 1.2.4.1. To assist reading comprehension by providing a focus on certain concepts prior to completing the Reading sections.
- 1.2.4.2. To evaluate understanding of relevant concepts after completing the Readings.
- 1.2.4.3. To promote active discussions between the trainer, trainee and trainee's co-workers using the questions as a starting point.
- 1.2.4.4. To document comprehension and/or application of objectives.
- 1.2.4.5. Written answers to these questions will be maintained in the training notebook as documentation of training.

1.2.5. The *Practical Exercises* will reinforce concepts from the study/discussion exercises and will teach "hands on"skills necessary for the analysis of clandestine laboratories.

2. HISTORY OF METHAMPHETAMINE

2.1. Objectives

2.1.1. To become familiar with the history of methamphetamine use and illicit manufacture.

2.1.2. To become aware of the changing trends in illicit manufacture of methamphetamine over time.

2.1.3. To understand the current and prior federal and state statutes pertaining to ephedrine, pseudoephedrine, phenylpropanolamine and other listed chemicals used in the illicit manufacture of controlled substances.

2.1.4. To become familiar with regulated chemicals.

2.2. Required Readings

2.2.1. Washington Administrative Codes (http://apps.leg.wa.gov/wac/): WAC 246-887-150 (schedule II immediate precursors); WAC 246-889 (precursor substance control).

2.2.2. Revised Code of Washington (http://apps.leg.wa.gov/rcw/): RCW 69.43 (precursors); RCW 69.50 (Uniform Controlled Substances Act); RCW 69.50.101 (definitions); RCW 69.50.440 (possession with intent to manufacture); RCW 69.50.511 (cleanup of hazardous materials related to manufacturing).

2.2.3. Federal Regulations (http://www.deadiversion.usdoj.gov/21cfr/index.html): Code of Federal Regulations, Title 21, Part 1300, Section 1300.02 (definitions relating to listed chemicals); http://www.deadiversion.usdoj.gov/schedules/index.html (list I and II chemicals).

2.2.4. Lukas, SE. The Encyclopedia of Psychoactive Drugs: Amphetamines. Chelsea House. 1985

2.3. Suggested Readings

2.3.1. Ogata A: Constitution of ephedrine – Desoxyephedrine; J Pharm Soc Jpn 451:751; 1919.

2.3.2. Inaba, D and Cohen W. Uppers, Downers, All Arounders 5th Edition. CNS Productions, Inc. 2004

2.4. Study/Discussion Exercises

2.4.1. Discuss the history of clandestine methamphetamine manufacture and include information on changes that have occurred as a result of legislation or actions by law enforcement. Suggestion: A timeline may be useful.

2.4.2. Explain the current definition of manufacturing as defined in the state and federal regulations.

2.4.3. Discuss the placement of pseudoephedrine on the Federal and State lists of Controlled Substances.

2.4.4. What are the definitions of List 1 and List 2 chemicals? Obtain copies of the List 1 and List 2 chemicals.

2.4.5.What are the current restrictions on the sale and purchase of ephedrine and pseudoephedrine?

3. INTRODUCTION TO CLANDESTINE LABORATORY ANALYSIS

3.1. **Objectives**

3.1.1. To develop an understanding of the types of hazards present in a clandestine drug laboratory.

3.1.2. To be familiar with the chemical hazards of evidence recovered from a clandestine laboratory and how to handle this evidence safely in the laboratory.

3.1.3. To become aware of the variety of methods available for the manufacture of controlled substances at a clandestine laboratory.

3.1.4. To be able to recognize common glassware and household supplies associated with clandestine laboratories.

3.1.5. To be able to select efficient and accurate methods for the analysis of clandestine laboratory samples.

3.2. **Required Readings**

3.2.1. Clandestine Lab Basic Guide. CLIC 12th Annual Training Seminar. New Orleans, LA

3.2.2. Christian, Donnell R., Forensic Investigation of Clandestine Laboratories, CRC Press, Boca Raton, 2004.

3.2.3. Willers-Russo, L. J., “Three Fatalities Involving Phosphine Gas, Produced as a Result of Methamphetamine Manufacturing,” Journal of Forensic Sciences, Vol. 44, No. 3, May 1999, pp. 647-652.

3.2.4. Cameron, M., “Iodine: Inhalation Hazards, Detection and Protection,” Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 12, No. 4, October 2002, pp. 18-19.

3.3. **Suggested Readings**

3.3.1. Jack B. Nimble, “The Construction and Operation of Clandestine Drug Laboratories,” Loompanics Unlimited, 1986, pp. 1-51.

3.3.2. Willers, L. J., “The Detection of Phosphine Gas Produced from Hydriodic Acid and the Evaluation of Detection Instruments for use in the Clandestine Laboratory Environments,” Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 13, No. 2, April 2003, pp. 14-24.

3.3.3. Uncle Fester, Secrets of Methamphetamine Manufacture (Editions 4, 5 and 7)

3.3.4. Drug Yield Calculator and associated references.

3.3.5. Keith Norman Synthesis Disk

3.4. **Study/Discussion Exercises**

3.4.1. Review the laboratory's PowerPoint presentations on clandestine laboratory recognition and processing. These may vary between labs but generally cover the common methods of manufacturing methamphetamine, safety, processing and sampling. They also may have an assortment of pictures showing actual clandestine laboratories encountered.

3.4.2. Review the Material Safety Data Sheets (MSDS) for the common chemicals found in a clandestine laboratory, including red phosphorus, iodine, hydriodic acid, lithium, sodium, anhydrous ammonia, sodium hydroxide, hydrochloric acid and some common solvents like methanol, toluene, xylene, etc.

3.4.3. List some common chemicals found in clandestine laboratories.

3.4.4. Identify some of the common chemicals that are incompatible.

3.4.5. Use the internet to research what kinds of recipes are available to someone who wants to manufacture methamphetamine.

3.4.6. Review the Clandestine Laboratory Investigating Chemist's Association's CLIC List to observe trends.

4. EPHEDRINE AND PSEUDOEPHEDRINE

4.1. Objectives

4.1.1. To develop an understanding of the role of ephedrine and pseudoephedrine in the clandestine manufacture of methamphetamine.

4.1.2. To learn how to analyze and identify chemicals involved in precursor extraction and isolation.

4.1.3. To learn about other active ingredients combined with pseudoephedrine and how they are affected by the reaction method employed.

4.1.4. To learn clandestine manufacturing methods of synthesizing pseudoephedrine or ephedrine.

4.2. Required Readings

4.2.1. Andrews, K. M., "Ephedra's Role as a Precursor in the Clandestine Manufacture of Methamphetamine," Journal of Forensic Sciences, Vol. 40, No. 4, July 1995, pp. 551-560.

4.2.2. Oulton, S. R., and Skinner, H. F., "Reaction Byproducts of Common Cold Tablet Ingredients Via Hydriodic Acid / Red Phosphorus," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 9, No. 4, October 1999, pp. 21-35.

4.2.3. Oulton, S. R., "Separation and Identification of Ephedrine, Pseudoephedrine and Methamphetamine Mixtures," Microgram, Vol. 30, No. 12, December 1997, pp. 289-296.

4.2.4. Melgoza, L., "Impurities in Methamphetamine Manufacture From Over-the-counter Pseudoephedrine Tablet Preparations," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 9, No. 2-3, April-July 1999, pp. 21-28.

4.2.5. Jacobs, J. L., Martinez, F. S., and Skinner, H. F., "Extraction of Reaction By-products of Common Cold Tablet Ingredients Via Hydriodic Acid Reduction," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 13, No. 1, January 2003, pp. 13-17.

4.3. Suggested Readings

4.3.1. Northrop, D., Knops, L., and Person, C., "Methamphetamine Manufacture From Cold and Allergy Medications Containing pseudoephedrine in Multi-Ingredient, Liquid, and Softgel Preparations," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 15, No. 2, April 2005, pp. 11-19.

4.3.2. DiPari, S. C., Bordelon, J. A., and Skinner, H. F., "Desloratadine: the Reaction Byproduct of the Reduction of Cold Tablets Containing Loratadine With Hydriodic Acid-Red Phosphorus," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 15, No. 1, January 2005, pp. 4-11.

4.3.3. Bentley, S., "The Plant They Call Ephedra," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 4, No. 4, October 1994, pp. 19-21.

4.3.4. Wallace, K., “Separation of Ephedra Alkaloids by GC/MS,” Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 12, No. 2, April 2002, pp. 14-16.
4.3.5. Allen. A. C. and Kiser, W. O., “Methamphetamine from Ephedrine: I. Chloroephedrines and Aziridines,” Journal of Forensic Sciences, Vol. 32, No. 4, July 1987, pp. 953-962.

4.3.6. Barker, W.D. and Antia, U., “A Study of the Use of Ephedra in the Manufacture of Methamphetamine,” Forensic Science International, Vol. 166, Issues 2-3, March 2007, pp. 102-109.

4.4. **Study/Discussion Exercises**

4.4.1. Discuss the ways in which pseudoephedrine may be extracted from various over-the-counter preparations and if an extraction is even needed.

4.4.2. Discuss how pseudoephedrine and ephedrine may be differentiated analytically.

4.4.3. Discuss other active ingredients commonly combined with pseudoephedrine and how they are affected by reaction conditions.

4.4.4. Discuss the formation and significance of oxazolidine compounds.

4.4.5. Is there a way to determine whether commercial grade pseudoephedrine/ephedrine or the plant Ephedra was used to manufacture methamphetamine?

4.4.6. Research the methods by which pseudoephedrine and ephedrine are manufactured both commercially and clandestinely.

4.5. **Practical Exercises**

4.5.1. Extract pseudoephedrine from tablets using both a solvent/filtration method and a base extraction. Save isolated pseudoephedrine for use in subsequent training modules.

4.5.2. Record the mass of the pseudoephedrine as it will be used later for yield determination.

4.5.3. Analyze the isolated pseudoephedrine by FTIR and GC/MS.

4.5.4. Analyze the tablet excipient material using the following methods (if available): FTIR, RAMAN, GC/MS, and microscopic.

4.5.5. Extract pseudoephedrine from a multi-ingredient cold and allergy preparation.

4.5.6. Isolate pseudoephedrine for IR from a methamphetamine/pseudoephedrine mixture.

5. METHAMPHETAMINE VIA THE HYDRIODIC ACID REACTION

5.1. **Objectives**

5.1.1. To develop an understanding of the hydriodic acid reduction of ephedrine/pseudoephedrine to methamphetamine.

5.1.2. To become aware of the variations of this reaction.

5.1.3. To be able to recognize the stages or steps of these reactions, typical by-products and waste materials.

5.1.4. To learn to analyze samples from these reactions and be able to determine from which step of the reaction the sample originated.

5.1.5. To learn techniques for distinguishing between acids commonly associated with clandestine methamphetamine manufacture: hydriodic, hydrochloric, sulfuric and hypophosphorus.

5.1.6. To understand the minimum testing required when reporting components of these methods of manufacture.

5.2. **Required Readings**

5.2.1. Skinner, H., "Methamphetamine Synthesis via Hydriodic Acid/Red Phosphorous Reduction of Ephedrine," Forensic Science International, Vol. 48, 1990, pp. 123-134.

5.2.2. Cantrell, T. S., John, B., Johnson, L., and Allen, A. C., "A Study of Impurities found in Methamphetamine Synthesized from Ephedrine," Forensic Science International, Vol. 39, 1988, pp. 39-53.

5.2.3. Allen. A. C. and Kiser, W. O., "Methamphetamine from Ephedrine: I. Chloroephedrines and Aziridines," Journal of Forensic Sciences, Vol. 32, No. 4, July 1987, pp. 953-962.

5.2.4. Oulton, S. and Skinner, H., "Identification of Common Inorganic Acids Encountered at Clandestine Laboratories", Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 8, No. 4, October 1998.

5.2.5. McKibben, T., "Analysis of Inorganic Components Found in Clandestine Drug Laboratory Evidence," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 5, No. 4, October 1995, pp. 19-33.

5.2.6. Heegel, R., et al., "Abbreviated Reaction Times in the Red Phosphorus-Iodine Manufacturing Method", Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 14, No. 3, July 2004, p 11.

5.2.7. Cameron, Mark, "Iodine: Inhalation Hazards, Detection and Protection" Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 12, No. 4, October 202, pp. 18-19.

5.2.8. Willers, L., "The Detection of Phosphine Gas Produced from Hydriodic Acid and the Evaluation of Detection Instruments for Use in Clandestine Laboratory Environments," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 13, No. 2, April 2003, pp. 14-24.

5.2.9. Vallely, P., A Single Step Process for Methamphetamine Manufacture Using Hypophosphorus Acid", Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 5, No. 2, April 1995, pp. 14-15.

5.2.10. Person, E.C., Knops, L.A., Heegel, R.A., and Northrop, D.M., "Phosphorus-Containing Reducing Agents: A review of their chemistry and use in the manufacture of methamphetamine and the significance of observed phosphate, phosphite, and hypophosphite in clandestine laboratory casework.", J. of Clandestine Laboratory Investigating Chemists' Association, 2006, Vol. 18, No. 2, April 2008, pp. 7-44.

5.2.11. Mayo, Erina et al., "The Reduction of Psuedoephedrin to Methamphetamine using Phosphorus Acid and Iodine" Journal of the Clandestine Laboratory Investigating Chemists Association Volume 19, Number 3, July 2009, pp. 30-39.

5.2.12. MacFarlane, K.J., and Neely, I.J. "Identification of Hypophosphorous and Hydrochloric Acid Mixtures Encountered at Clandestine Laboratories", Journal of the Clandestine Laboratory Investigating Chemists Association Volume 19, Number 2, April 2009, pp. 16-35.

5.2.13. Appendix A: IODINE

5.2.14. Appendix B: RED PHOSPHORUS

5.2.15. Appendix C: ACIDS

5.3. **Suggested Readings**

5.3.1. Skinner, H. F. and Oulton, S. R., "Identification and Quantitation of Hydriodic Acid Manufactured from Iodine, Red Phosphorus and Water," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 5, No. 4, October 1995, pp. 12-18.

5.3.2. Massetti, J., "Central California Mexican National Drug Lab Trends: 1994," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 4, No. 3, July 1994, pp. 13-15.

5.3.3. Largent, D., "An Overview of the Mexican National Lab Situation," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 4, No. 2, April 1994, pp. 30-35.

5.3.4. Bentley, S., "A Validation Study of the "Cold Method", Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 3, No. 1, January 1993, pp. 12-13.

5.3.5. Chappell, J. and Lee, M., "Isolation and Identification of Methamphetamine Hydriodide from Clandestine Laboratory Samples", Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 5, No. 2, April 1995, pp. 16-20.

5.3.6. Popovich, G.L., "Instant Methamphetamine", Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 5, No. 3, July 1995, pp. 7-8.

5.3.7. Vallely, P., “An efficient Method for the Synthesis of Hydriodic Acid from Hydrogen Sulphide”, Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 13, No.2, April 1995, pp. 21-22.

5.3.8. Windahl, K.L. et al, “Investigation of the Impurities Found in Methamphetamine Synthesized from Pseudoephedrine by Reduction with Hydriodic Acid and Red Phosphorus”, Forensic Science International, Vol. 76, 1995, pp. 97-114.

5.3.9. Oulton, S. and Skinner, H., “Reaction Byproducts of Common Cold Tablet Ingredients via Hydriodic Acid/Red Phosphorus”, Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 9, No. 4, October 1999, pp. 21-35.

5.3.10. Skinner, H. and Jacobs, J., “Extraction of Reaction By-Products of Common Cold Tablet Ingredients Via Hydriodic Acid Reduction”, Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 13, No. 1, January 2003, pp. 13-14.

5.3.11. Abercrombie, J. Thomas, “Empirical Study of the Effects of Initial Precursor Amount in Regard to Final Yield, Ratio of By-Products and Other Information in the Ephedrine/HI/Red Phosphorus Synthetic Route”, 1st Annual Technical Training Seminar, Clandestine Laboratory Investigating Chemists Association, 1991.

5.3.12. Jones, Arthur, et al., “Enantioseparation Of Methylamphetamine By Capillary Electrophoresis: A Survey Of MethylamphetamineSamples Seized In The Act” Journal of the Clandestine Laboratory Investigating Chemists Association Volume 21, Number 3, July 2011 pp.9-11.

5.3.13. Walker, Angela et al., “Phosphorous Acid Flakes Used As A Substitute For Red Phosphorus In The Reduction Of (Pseudo)Ephedrine To Methamphetamine”,Journal of the Clandestine Laboratory Investigating Chemists Association Volume 20, Number 2, April 2010 pp.14-18.

5.4. **Study/Discussion Exercises**

5.4.1. List all of the different methods (variations) of manufacturing methamphetamine using the hydriodic acid reduction method.

5.4.2. Review the red phosphorus - hydriodic acid - ephedrine/pseudoephedrine reduction mechanism.

5.4.2.1. Discuss each of the steps that are used and note the common modifications and variations that have been seen in the past.

5.4.2.2. Discuss the purpose of each ingredient, noting any possible substitutions that can be used.

5.4.2.3. Is red phosphorus a necessary part of this reaction? What are the sources of red phosphorus? Can other chemicals be used in place of red phosphorus? If so, what are they?

5.4.2.4. Discuss the analysis of the common precursors and reagents to this reaction.

5.4.2.5. Research and discuss possible sources for elemental iodine.

5.4.2.6. Create a reference standard of substituted naphthalenes (232 compounds) using phenyl-2-propanone and hydriodic acid and perform a GC/MS analysis. Keep this reference standard for use in casework.

5.4.3. Review the hypophosphorous acid – ephedrine/pseudoephedrine reduction mechanism.

5.4.3.1. Research and describe a recipe for this method and discuss the different steps involved.

5.4.3.2. How could you differentiate between this method and the red phosphorus – hydriodic acid method?

5.4.4. Discuss the safety issues related to the hydriodic acid reaction. What is the most dangerous step in this reaction?

5.4.5. How would you explain the presence of phenyl-2-propanone in a sample of methamphetamine that was produced by the hydriodic acid method?

5.4.6. List the common non-controlled ingredients of this reaction. How many tests are required for their identification? List possible tests that can be used for at least four non-controlled ingredients.

5.5. **Practical Exercises**

5.5.1. Draw a flow chart, describing all of the steps involved in the red phosphorus - hydriodic acid method of methamphetamine synthesis. At each step note the pH, the number of phases and whether each phase is water soluble or not.

5.5.2. Extract red phosphorus from matchbook striker plates and perform a complete analysis using as many of the techniques listed in Appendix B that are available.

5.5.3. Conduct a search to find an internet recipe for precipitating elemental iodine from a strong tincture solution (7%). Perform the precipitation reaction on a small scale. Analyze a small amount of your collected iodine using as many techniques listed in Appendix A that are available (make sure to follow the correct procedure).

5.5.4. Research the ratios of pseudoephedrine/iodine/red phosphorus/water commonly used to manufacture methamphetamine. Consult with your trainer before deciding on the proportions you will use. Make methamphetamine using your extracted pseudoephedrine from 43.5.1. Record the mass/volume of all reactants and final product to be used later for yield determinations.

5.5.4.1. Collect aliquots at various times during the reaction. Analyze for the presence of reactants, final product and by-products.

5.5.4.2. Save the red phosphorus that you filter out and perform a complete analysis. Hint: try a variety of extractions/solvents to isolate phenyl-2-propanone, methamphetamine, and substituted naphthalenes. What would you conclude from your findings?

5.5.4.3. After basifying and extracting with a non-polar solvent, collect some of the organic and aqueous layers and perform a complete analysis. Try to isolate and analyze iodine from the aqueous layer.

5.5.5. Try dissolving elemental iodine in numerous solvents such as: water, methanol, petroleum ether, chloroform and hexane. Note color, if any, of the resulting solution.

5.5.6. Using reagent-quality acids, carry out the precipitation tests for hydriodic, hydrochloric, sulfuric and hypophosphorus acids (See Appendix E 24~~2~~.4.2). If available, try the tests also with clandestinely-produced HI.

6. METHAMPHETAMINE VIA THE DISSOLVING METAL / AMMONIA REDUCTION

6.1. Objectives

6.1.1. To develop an understanding of metal reduction reactions that can be used in the clandestine manufacture of methamphetamine.

6.1.2. To learn to analyze samples from these reactions and be able to determine what step of the reaction the sample originated.

6.1.3. To learn how ammonia can be generated and used in this reaction.

6.1.4. To develop an understanding of the special safety concerns related to this process.

6.2. Required Readings

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

6.2.2. Dawson, N., "The Sodium - Ammonia 'Nazi' Method of Methamphetamine Synthesis: An Historical Overview, Methodology and Case Reviews," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 5, No. 3, July 1995, pp. 12-14.

6.2.3. Person, E.C., Meyer, J.A., and Vyvyan, J.R., "Structural Determination of the Principal Byproduct of the Lithium-Ammonia Reduction Method of Methamphetamine Manufacture," Journal of Forensic Sciences, Vol. 50, No. 1, January 2005, pp. 87-95.

6.2.4. Person, E., Knops, L. A., Northrop, D. M., and Sheridan, S. P., "One-Pot Methamphetamine Manufacture," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 14, No. 2, April 2004, pp. 14-15.

6.2.5. Person, E. "Clandestine Ammonia Generation," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 14, No. 1, January 2004, pp. 20-27.

6.2.6. Worley, D., "Evaluation of Ammonium Test Paper," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 12, No. 2, April 2002, pp. 17-19.

6.2.7. Smiley, J.C., Hickmon, T., and Karr, C., "Analysis of Anhydrous Ammonia Via Precipitation of Ammonium Salt," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 11, No. 1, January 2001, pp. 31-34.

6.2.8. Anderson, O.C., "Lithium Spot Test," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 10, No. 3, July 2000, p. 11.

6.2.9. McKibben, T., "Analysis of Inorganic Components Found in Clandestine Drug Laboratory Evidence," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 5, No. 4, October 1995, pp. 19-33.

6.3. Suggested Readings

6.3.1. Kummerlowe, D., "Initial Considerations for Handling 5-Gallon Pressurized Tanks of Ammonia Gas Associated with Clandestine Drug Labs," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 6, No. 4, October 1996, pp. 23-030.

6.3.2. "Methamphetamine Byproduct from Birch Reduction Tentatively Identified," Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 7, No. 2, April 1997, pp. 7-10.

6.3.3. Kelly, C. A., Lawrence, D. S., Murray, G. M., and Uy, O. M., "Methamphetamine Synthesis Inhibition: Dissolving Metal Reduction." Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 12, No. 3, July 2002, pp. 10-15.

6.3.4. Birch, A.J., "Reduction by Dissolving Metals, Part II," Journal of the Chemical Society, June 1945, pp 809-813.

6.3.5. View WSP's videos on ammonia generation and the "One Pot" method.

6.4. Study/Discussion Exercises

6.4.1. Review the metal reduction reactions that can be used to make methamphetamine.

6.4.2. Discuss each of the steps that are used and note the common modifications and variations that may be encountered.

6.4.3. Discuss the safety issues related to this reaction.

6.4.4. Discuss the analysis of the common precursors and reagents used in this reaction.

6.4.5. Discuss byproducts that are unique to the metal reduction process. Under what conditions are they formed?

6.4.6. Discuss what chemical compounds might be present in the reaction sludge from a lithium/ammonia reduction process and how one might analyze and identify each.

6.5. Practical Exercises

6.5.1. Cook methamphetamine starting from lithium or sodium, liquefied ammonia and pseudoephedrine. Collect samples at various times during the reaction. Analyze for the presence of reactants, final product and by-products. Record the mass/volume of all reactants for use later for yield determinations.

6.5.2. Generate and condense ammonia gas using an ammonium-based fertilizer and sodium hydroxide (can combine with exercise 6.5.1).

6.5.3. Draw a flow chart, describing all of the steps involved in the process. At each step note the pH, key chemical compounds present, the number of phases and whether each phase is water soluble or not.

6.5.4. (Optional) Perform the same cook as above utilizing the conditions to optimize the formation of the CMP compound.

7. OTHER METHODS OF METHAMPHETAMINE SYNTHESIS

7.1. **Objectives**

7.1.1. To develop an understanding of the various reactions that historically were used to manufacture methamphetamine and phenyl-2-propanone, a precursor used in the clandestine manufacture of methamphetamine.

7.1.2. To be able to recognize what type of reaction was utilized in the manufacture of methamphetamine.

7.1.3. To become aware of the variations of these reactions.

7.1.4. To be able to recognize the stages or steps of the reaction used.

7.1.5. To learn to analyze samples from the reaction used and be able to determine from what step of the reaction the sample originated.

7.2. **Required Readings**

7.2.1. Weaver, K. and Young, E., “An Analyst’s Guide to the Investigation of Clandestine Laboratories,” Health Canada, June 1995, pp. 4-1 to 4-4.

7.2.2. Allen, A. C., Stevenson, M. L., Nakamura, S. M. and Ely, R. E., “Differentiation of Illicit Phenyl-2-Propanone Synthesized from Phenylacetic Acid with Acetic Anhydride Versus Lead (II) Acetate,” Journal of Forensic Sciences, Vol. 37, No. 1, Jan. 1992, pp. 301-322.

7.2.3. Uncle Fester, “The Secrets of Methamphetamine Manufacture,” Loompanics Unlimited, 1987, pp. 11-25.

7.2.4. Ama, Verweij, “Impurities in Illicit Drug Preparations: Amphetamine and Methamphetamine,” Forensic Science Review, Vol. 1, No. 1, 1989, pp. 2-111.

7.2.5. Allen, A. C. and Cantrell, T. S., “Synthetic Reductions in Clandestine Amphetamine and Methamphetamine Laboratories: A Review,” Forensic Science International, 42, 1989, pp. 183-2192.

7.2.6. Wassink, B. H., Duijndam, A., Jansen, A. C. A., ”A Synthesis of Amphetamine,” Journal of Chemical Education, Vol. 51, 1974, p. 671.

7.2.7. CLIC 3rd Annual Technical Training Seminar “A Review of the Synthesis and Analysis of P2P, Amphetamine and Methamphetamine” Vol. 1 and Vol. 2., 1993.

7.2.8. Allen A., Kiser W. “Methamphetamine from Ephedrine: 1. Chloroephedrines and Aziridines” Journal of Forensic Sciences, 32, 1987, pp. 953-962.

7.2.9. California Criminalistics Institute Lecture Notes, “Clandestine Laboratory Analysis and Synthesis” Chapters 2 and 3, 1997.

7.2.10. Norman, Keith, “The Synthesis Of Amphetamine And Methamphetamine: A “Big” Picture” Journal of the Clandestine Laboratory Investigating Chemists Association Volume 19, Number 3, July 2009, pp. 10-29.

7.2.11. Bozenko, Joseph S Jr. “Comparisons of Mexican Methamphetamine Laboratories” CLIC Technical Training Seminar 2011.

7.3. **Suggested Readings**

7.3.1. Herbst, R. M. and Manske, R. H., “Methyl Benzyl Ketone”, Organic Synthesis, Collective Volume 2, pp. 389-392.

7.3.2. Forbes, I. J. and Kirkbride, K. P., “The Origin of Alkenes in Illicit Amphetamine: An Examination of the Illicit Synthesis of Phenyl-2-Propanone,” Journal of Forensic Sciences, Vol. 37, No. 5, 1992, pp. 1311-1318.

7.3.3. Dal Cason, T. A., “Some Information Regarding Phenyl-2-Propanone, “Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 4, No. 1, January 1994, pp. 21-23.

7.3.4. Dal Cason, T. A., Angelos, S. A. and Raney, J. K., “A Clandestine Approach to the Synthesis of Phenyl-2-Propanone from Phenylpropenes,” Journal of Forensic Sciences, Vol. 29, No. 4, 1984, pp. 1187-1208.

7.3.5. Ely, R. A., “Synthesis and Reductions of 1-Phenyl-2-Nitropropene to Phenyl-2-Propanone or Amphetamine,” Personal communication, 1991.

7.3.6. Karg, E., Marcus, F., “Preperation of Arylalkylamines” Ber, 75B, 1942, pp1850-1854.

7.3.7. Crossley, Moore, “Studies of the Leuckart Reaction”, Journal of Organic Chemistry, 9, 1994, pp. 529-536.

7.3.8. Bozenko, Joseph S Jr. “ Clandestine Enantiomeric Enrichmentof d–Methamphetamine via Tartaric Acid Resolution” Journal of the Clandestine Laboratory Investigating Chemists Association Volume 18, Number 3, July 2008, pp. 4-8.

7.4. **Study/Discussion Exercises**

7.4.1. Review the reactions that can be used to make phenyl-2-propanone: phenylacetic acid and lead (II) acetate; phenylacetic acid, acetic anhydride and sodium acetate or pyridine; sodium metal, benzyl cyanide alpha-phenylacetonitrile, ethyl acetate and sodium ethoxide; phenylacetic acid, acetic acid and thorium nitrate. Discuss the similarities and differences of each method. Identify any unique by-products that can be used to identify which method was used.

7.4.2. Review the methods of the synthesis of the precursors to phenyl-2-propanone and methamphetamine, phenylacetic acid, benzyl cyanide and methylamine hydrochloride.

7.4.3. Review the reactions that can be used to synthesize methamphetamine using phenyl-2-propanone and methylamine: aluminum and mercuric chloride method; formic acid/formamide (Leuckart Reaction); sodium metal or platinum oxide catalyst; sodium cyanodihydriborate reductive

amination via an imine. Discuss the similarities and differences of each method. Identify any unique by-products that can be used to identify which method was used. Discuss each of the steps that are used and note the common modifications and variations that have been seen in the past. Discuss the purpose of each ingredient, noting any possible substitutions that can be used. Discuss the safety issues related to these reactions.

7.4.4. Review the laboratory file and/or conduct an internet search for clandestine recipes for the manufacture of phenyl-2-propanone and methamphetamine using phenyl-2-propanone.

7.4.5. Review the non-chiral methods of synthesizing methamphetamine using ephedrine and/or pseudoephedrine: thionyl chloride or phosphorus pentachloride; direct hydrogenation using a perchloric acid and palladium catalyst. What by-products and potential intermediary products can be formed using these methods? Discuss the similarities and differences of each method.

7.5. **Practical Exercises**

7.5.1. Your trainer will pick three synthesis methods. Draw a flow chart, describing all of the steps involved in each process. At each step note the pH, the number of phases and whether each phase is water soluble or not.

7.5.2. Analyze the following common precursor materials using as many pertinent techniques as possible (ie.odor, pH, X-ray, GC/MS, FT-IR, color tests, etc): Phenylacetic acid, acetic anhydride, sodium acetate, phenyl-2-propanone, methylamine, mercuric chloride, aluminum foil, lead acetate.

8. METHCATHINONE

8.1. Objectives

8.1.1. To develop an understanding of methcathinone synthesis methods.

8.1.2. To learn to analyze samples from these reactions.

8.1.3. To become aware of the variations of these reactions.

8.1.4. To be able to recognize the stages or steps of the reaction used.

8.2. Required Readings

8.2.1. Zhingel, K., et. al., "Ephedrone: 2-Methylamino-1-Phenylpropan-1-One (Jeff)", J Forensic Sci, Vol. 36, No. 3, pp 915-920.

8.2.2. Methcathinone postings and references available on the Keith Norman Disk.

8.2.3. Uncle Fester, "The Secrets of Methamphetamine Manufacture," Loompanics Unlimited, Chapter 16.

8.3. Suggested Readings

8.3.1. Berrang, B., Lewin, A., Carroll, F., "Enantiomeric α-Aminopropiophenones (Cathinone): Preparation and Investigation," J Org Chem, Vol. 47, Iss. 13, 1982, pp 2643-47.

8.4. Study/Discussion Exerices

8.4.1. Discuss what impurities or by-products might be present from the manufacture of methcathinone. Explore the formation of pyrazine compounds through dimerization of methcathinone.

8.4.2. Discuss what similarities might exist between samples taken from a methcathinone lab and a methamphetamine lab?

8.4.3. Search the internet for possible methcathinone analogs and familiarize yourself with the synthesis.

8.5. Practical Exercises

8.5.1. Synthesize methcathinone using pseudoephedrine or ephedrine as a precursor and an oxidizer. Obtain analytical data from various steps of the process.

8.5.2. Draw out the reaction steps of manufacturing methcathinone via Jones Oxidation and Sarett Oxidation.

9. 3,4-METHYLENEDIOXYAMPHETAMINE (MDA) AND 3,4-METHYLENEDIOXYMETHAMPHETAMINE (MDMA)

9.1. **Objectives**

9.1.1. To develop an understanding of the various reactions that are used to manufacture 3,4-methylenedioxyamphetamine (MDA) and 3,4-methylenedioxymethamphetamine (MDMA).

9.1.2. To be able to recognize what type of reaction was utilized in the manufacture of MDA or MDMA.

9.1.3. To become aware of the variations of these reactions.

9.1.4. To be able to recognize the stages or steps of the reaction used.

9.1.5. To learn to analyze samples from the reaction used and be able to determine from what step of the reaction the sample originated.

9.2. **Required Readings**

9.2.1. Noggle, F. et al, "GC and MS Analysis of Samples from a Clandestine Laboratory Involved in the Synthesis of Ecstacy from Sassafras Oil" Journal of Chromatographic Science, Vol. 29, No. 99, 1991, pp. 168-174.

9.2.2. "Structure-Activity Relationships, Syntheses, Precursor Preparation and Analyses of Methylenedioxyamphetamine and its Analogs and Homologs", Fourth Annual Technical Training Seminar, Clandestine Laboratory Investigating Chemists Association, Volumes I, II, III, and IV, 1994.

9.2.3. Dal Cason, Terry A., "The Characterization of Some 3,4-Methylenedioxyphenylisopropylamine (MDA) Analogs", Journal of Forensic Sciences, Vol. 34, No. 4, July 1989, pp. 928-961.

9.2.4. Lukaszewski, Theodore, "Spectroscopic and Chromatographic Identification of Precursors, Intermediates, and Impurities of 3,4-Methylenedioxyamphetamine Synthesis", Journal of the Association of Official Analytical Chemists, Vol. 61, No. 4, 1978, pp. 951-967.

9.2.5. Bailey, Keith, et al., "Identification of the N-methylated Analogs of the Hallucinogenic Amphetamines and Some Isomers", Journal of the Association of Official Analytical Chemists, Vol. 58, No. 1, 1975, pp. 62-69.

9.2.6. Kovacs, Edward III, and Kirby, Dean "Manufacture of3,4–Methylenedioxyamphetamine from Helional Using Beckmannand Hofmann Rearrangements" Journal of the Clandestine Laboratory Investigating Chemists Association Volume 23, Number 1, January 2013, pp. 5-14.

9.3. **Suggested Readings**

9.3.1. "Analytical Profiles of Substituted 3,4-methylenedioxyamphetamine: Designer Drugs related to MDA" CND Analytical, 1988.

9.3.2. "Analytical Profiles of Substituted 3,4-methylenedioxyamphetamine: Designer Drugs related to MDA" Volume II, CND Analytical, 1991.

9.3.3. "Analytical Profiles of Precursors and Essential Chemicals", CND Analytical, 1990.

9.3.4. "Analytical Profiles of the Hallucinogens", CND Analytical, 1991.

9.3.5. Strike, "Total Synthesis II", Panda Ink, San Antonio, TX 1999.

9.3.6. Uncle Fester, "Secrets of Methamphetamine Manufacture including recipes for MDA, Ecstasy, and other psychedelic amphetamines", 5th edition, Loompanics Unlimited, Port Townsend, WA, 1999.

9.3.7. Professor Buzz, "Recreational Drugs" Loompanics Unlimited, Port Townsend, WA 1989.

9.3.8. Alexander and Ann Shulgin, "PIHKAL", Transform Press, Berkeley, CA 1991.

9.3.9. Lang, Richard "Hallucinogens A Forensic Drug Handbook", Academic Press, London, 2003 pages 139-190.

9.4. **Study/Discussion Exercises**

9.4.1. Go through the Keith Norman Disk for MDMA and MDA. Review the reactions that can be used to make MDA and MDMA, read the descriptions on how to manufacture MDA or MDMA by the various reaction methods.

9.4.2. Review the laboratory file and/or conduct an internet search for clandestine recipes for the manufacture of MDA and MDMA.

9.5. **Practical Exercises**

9.5.1. Your instructor will chose three of the methods of MDMA or MDA synthesis. For each method draw a flow chart, describing all of the steps involved in each process. At each step note the pH, the number of phases and whether each phase is water soluble or not.

9.5.2. Analyze as many of the following common precursor materials using as many pertinent techniques as possible (ie. pH, X-ray, GC/MS, FT-IR, color tests, etc): 4-methylenedioxyphenyl-2-propanone (MDP-2-P), Isosafrole, Safrole, formamide, ammonium formate, piperonal, nitroethane, vanillin, cuprous oxide, ammonium acetate, dibromomethane, bromosafrole, chlorosafrole, methylamine hydrochloride, mercuric bromide, mercuric chloride, sodium cyanoborohydride.

10. PHENCYCLIDINE (PCP)

10.1. **Objectives**

10.1.1. To develop an understanding of the various reactions that are used to manufacture phencyclidine (PCP).

10.1.2. To be able to recognize what type of reaction was utilized in the manufacture of PCP.

10.1.3. To become aware of the variations of these reactions.

10.1.4. To be able to recognize the stages or steps of the reaction used.

10.1.5. To learn to analyze samples from the reaction used and be able to determine from what step of the reaction the sample originated.

10.2. **Required Readings**

10.2.1. "A Review of the Syntheses and Analyses of Phencyclidine and its Analogs" Sixth Annual Technical Training Seminar, Clandestine Laboratory Investigating Chemists Association, 1996.

10.2.2. Angelos, S. et al, "The Identification of Unreacted Precursors, Impurities, and By-Products in Clandestinely Produced Phencyclidine Preparations" Journal of Forensic Sciences, Vol. 35, No. 6, 1990, pp. 1297-1302.

10.2.3. All of the articles found in the PCP section of the seized drugs training manual.

10.3. **Suggested Readings**

10.3.1. "Analytical Profiles of Precursors and Essential Chemicals", CND Analytical, 1990.

10.3.2. "Analytical Profiles of the Hallucinogens", CND Analytical, 1991.

10.3.3. Professor Buzz, "Recreational Drugs" Loompanics Unlimited, Port Townsend, WA 1989.

10.4. **Study/Discussion Exercises**

10.4.1. Review the reactions that can be performed to manufacture PCP, from the CLIC monograph: the Kalir Method, the Maddox Method and the Godefroi Method. Discuss the similarities and differences of each method. Identify any unique by-products that can be used to determine which method was used.

10.4.2. Review the methods for the synthesis of piperidine from pyridine.

10.4.3. Review the laboratory file and/or conduct an internet search for clandestine recipes for the manufacture of PCP.

10.5. **Practical Exercises**

10.5.1. For each method of PCP synthesis listed in the CLIC monograph, draw a flow chart, describing all of the steps involved in each process. At each step note the expected pH, the number of phases and whether each phase is water soluble or not.

10.5.2. Analyze as many of the following materials used in PCP manufacture, using as many pertinent techniques as possible (ie. pH, X-ray, GC/MS, FT-IR, color tests, etc): sodium metal, ethanol, ruthenium dioxide, piperidine, pyridine, cyclohexanone, potassium or sodium cyanide, 1-piperidinocyclohexane carbonitrile, bromobenzene, magnesium metal, sodium bisulfite, para-toluene-sulfonic acid.

11. LYSERGIC ACID DIETHYLAMIDE (LSD)

11.1. **Objectives**

11.1.1. To develop an understanding of the various reactions that are used to manufacture LSD.

11.1.2. To be able to recognize what type of reaction was utilized in the manufacture of LSD.

11.1.3. To become aware of the variations of these reactions.

11.1.4. To be able to recognize the stages or steps of the reaction used.

11.1.5. To learn to analyze samples from the reaction used and be able to determine from what step of the reaction the sample originated.

11.2. **Required Readings**

11.2.1. All of the articles found in the LSD section of the seized drugs training manual.

11.2.2. All of the method articles on LSD synthesis found in the Keith Norman Disk.

11.2.3. Alexander and Ann Shulgin, “TIHKAL:The Continuation”, Transform Press, Berkeley, CA 1997. pages 490-499.

11.2.4. “Analytical Profiles of Precursors and Essential Chemicals”, CND Analytical, 1990.

11.3. **Suggested Readings**

11.3.1. Uncle Fester, Practical LSD Manufacture, Loompanics Unlimited, Port Townsend, 1995.

11.3.2. “Analytical Profiles of the Hallucinogens”, CND Analytical, 1991.

11.3.3. Professor Buzz, “Recreational Drugs” Loompanics Unlimited, Port Townsend, WA 1989.

11.3.4. Cam Cloud, “LSD: Acid Trips and Chemistry”, Ronin Publishing Berkely, CA 1999 pages 93-121.

11.3.5. Lang, Richard “Hallucinogens A Forensic Drug Handbook”, Academic Press, London, 2003 pages 139-190.

11.4. **Study/Discussion Exercises**

11.4.1. Review the reactions that can be performed to manufacture LSD, from the Keith Norman Disk. Discuss the similarities and differences of each method. Identify any unique by-products that can be used to identify which method was used.

11.4.2. Review the methods for the synthesis of lysergic acid, found on the Keith Norman Disk.

11.4.3. Review the laboratory file and/or conduct an internet search for clandestine recipes for the manufacture of LSD.

11.5. **Practical Exercises**

11.5.1. For the following methods of LSD synthesis: the original patent by Albert Hoffman, and LSD from Ergotamine Tartrate from TiHKAL, draw a flow chart, describing all of the steps involved in each process. At each step note the pH, the number of phases and whether each phase is water soluble or not. (Note any special considerations, such as use of a dark room etc.)

11.5.2. Analyze as many of the following materials used in LSD manufacture, using as many pertinent techniques as possible (ie. pH, XRF or SEM, GC/MS, FT-IR, color tests, etc): alumina, diethylamine, dichloroethane, dimethyl formamide, ergot alkaloid, ergotamine tartrate, ethylene dichloride, hydrazine, lithium hydroxide, lysergic acid, N,N-Carbonyidiimidazole, sodium nitrite, anhydrous sodium sulfate, sulphur trioxide, and triethylamine.

12. TRYPTAMINES

12.1. **Objectives**

12.1.1. To develop an understanding of the reactions used to manufacture various types of tryptamines.

12.1.2. To be able to recognize what type of reaction was utilized in the manufacture of a tryptamine.

12.1.3. To become aware of the variations of these reactions.

12.1.4. To be able to recognize the stages or steps of the reaction used.

12.1.5. To learn to analyze samples from the reaction used and be able to determine from what step of the reaction the sample originated.

12.2. **Required Readings**

12.2.1. “Synthesis, Analog Synthesis, and Precursor Synthesis”, Tryptamines Monograph, Vol. 1, CLIC Sept 2001.

12.2.2. “Analytical Data and Natural Product Information”, Tryptamines Monograph, Vol. 2, CLIC Sept 2001.

12.2.3. Cowie, J.S., et. al., “Identification of the Major Impurities in the Illicit Manufacture of Tryptamines and Related Compounds”, Journal of Forensic Sciences, Vol. 27, No. 3, July 1982, pp. 527-540.

12.2.4. Tryptamine information and references available on the Keith Norman Disk.

12.2.5. Kirby, Dean, “Latest Trends in Hallucinogenic Tryptamine & Phenethylamine Analogs”, NWAFS Seminar, Nov. 14-18, 2005.

12.3. **Suggested Readings**

12.3.1. Shulgin and Shulgin, TiKHAL, the Continuation, Transform Press, Berkeley, CA 1991.

12.3.2. Psilocyn and psilocybin information and references available on the Keith Norman Disk.

12.3.3. Smith, T.A. “Tryptamine and Related Compounds in Plants”, Phytochemistry, 1977, 16, pp 171-5. Sundberg, R.J., Indoles, Academic Press, 1996.

12.3.4. Laing, Richard, Hallucinogens: A Forensic Drug Handbook, Academic Press, 2003, pp 99-105, 150-3.

12.4. **Study/Discussion Exercises**

12.4.1. Review indole chemistry and its application to the manufacture of tryptamines.

12.4.2. Discuss how one might obtain tryptophan and subsequently convert it to tryptamine.

12.4.3. Explore how tryptamines currently encountered in controlled substance casework might be synthesized via the information available in your reference materials.

12.5. **Practical Exercises**

12.5.1. Draw out the steps used to synthesize N-substituted tryptamines using oxalyl chloride. Indicate where and how modifications to the synthesis could produce dimethyltryptamine (DMT), diethyltryptamine (DET), and 5-methoxy-N,N-diisopropyltryptamine (Foxy).

12.5.2. Draw out the steps used to synthesize α-alkyltryptamines using 1-nitropropane. Indicate where and how modifications to the synthesis could produce α-methyltryptamine (AMT) and α-ethyltryptamine (AET).

12.5.3. Analyze as many of the precursors and reagents as possible found in the Chemical Reference Table on the Keith Norman Disk for α-ethyltryptamine, dimethyltryptamine, and diethyltryptamine.

13. PHENETHYLAMINE (PEA) ANALOGS

13.1. **Objectives**

13.1.1. To develop an understanding of the reactions used to manufacture various types of phenethylamines analogs.

13.1.2. To learn to analyze samples from these reactions.

13.1.3. To become aware of the variations of these reactions.

13.1.4. To be able to recognize the stages or steps of the reaction used.

13.2. **Required Readings**

13.2.1. Shulgin and Shulgin, PiKHAL, A Chemical Love Story, Transform Press, Berkeley, CA 1991, Entries: #20, 22-24, 32-34, 39-43, 62, 64, 66-68, 142.

13.2.2. Kirby, Dean, “Latest Trends in Hallucinogenic Tryptamine & Phenethylamine Analogs”, NWAFS Seminar, Nov. 14-18, 2005.

13.2.3. Phenethylamine information and references available on the Keith Norman Disk.

13.3. **Suggested Readings**

13.3.1. Laing, Richard, Hallucinogens: A Forensic Drug Handbook, Academic Press, 2003, pp 79-97.

13.4. **Study/Discussion Exercises**

13.4.1. Discuss which non-controlled phenethylamines have been encountered in casework and how they were identified.

13.4.2. Discuss how the Controlled Substance Analog Act would be applied to encountered phenethylamines and which controlled substances would be most similar structurally.

13.4.3. Explore how phenethylamines analogs currently encountered in controlled substance casework might be synthesized using the information available in your reference materials.

13.5. **Practical Exercises**

13.5.1. Draw out the steps used to synthesize 2,5-dimethoxyphenethylamines starting with 2,5-dimethoxybenzaldehyde. Indicate how modifications to the synthesis could produce both alkyl (e.g. 2C-E), halogenated (e.g. 2C-B), and sulfur (e.g. 2C-T-7) compounds at the 4-position.

13.5.2. Modify the syntheses created above to create DOM analogs instead.

13.5.3. Discuss the NBOMe series of phenethylamines and research possible synthetic methods that could be encountered.

13.5.4. Analyze as many of the precursors and reagents as possible found in the Chemical Reference Table on the Keith Norman Disk for 2,5-dimethoxyamphetamine, 4-bromo-2,5-dimethoxyphenethylamine, 4-ethyl-2,5-dimethoxyamphetamine, DOB, and STP.

13.5.5. Try to find precursor or finished product of these types of compounds being sold on the Internet.

14. GAMMA-HYDROXYBUTYRATE (GHB)

14.1. **Objectives**

14.1.1. To develop an understanding of gamma-hydroxybutyrate (GHB) synthesis methods.

14.1.2. To learn to analyze samples from these reactions.

14.1.3. To become aware of the variations of these reactions.

14.1.4. To be able to recognize the stages or steps of the reaction used.

14.2. **Required Readings**

14.2.1. Synthesis references provided in the GHB Monograph, presented at 2000 annual CLIC meeting.

14.2.2. Mercer, J.W., et. al., “Comparative Analysis of Gamma-Hydroxybutyrate and Gamma-Hydroxvalerate Using GC/MS and HPLC”, J Forensic Sci, Vol. 52, No. 2, pp 383-388.

14.2.3. Chappell, J., Meyn, A., and Ngim, K., “The Extraction and Infrared Identification of Gamma-Hydroxybutyric Acid (GHB) from Aqueous Solutions,” J Forensic Sci, Vol. 49, No. 1, pp 1-8.

14.3. **Suggested Readings**

14.3.1. Other references provided in the GHB Monograph, presented at 2000 annual CLIC meeting.

14.4. **Study/Discussion Exercises**

14.4.1. Discuss what impurities or by-products, if any, might be present from the manufacture of GHB.

14.4.2. Review the chemical interconversion between GHB and Gamma-butyrolactone (GBL) in aqueous solution as a function of pH.

14.4.3. Discuss the manufacturing process for GHB analogs such as Methyl-4-Acetoxybutanoate (MAB), trans-4-Hydroxy-Crotonic Acid (T-HCA), and gamma-Hydroxyvaleric acid (GHV).

14.5. **Practical Exercises**

14.5.1. Synthesize GBL from tetrahydrofuran (THF) using calcium hypochlorite. Obtain analytical data from the synthesis.

14.5.2. Synthesize both the sodium and potassium salts of GHB from GBL. Obtain analytical data from each synthesis.

15. FENTANYL AND ITS DERIVATIVES

15.1. Objectives

15.1.1. To develop an understanding of fentanyl synthesis methods, and understand which precursors can be modified to make productive analogs.

15.1.2. To learn to analyze samples from these reactions.

15.1.3. To become aware of other possible variations to these reactions.

15.1.4. To be able to recognize the stages or steps of the reactions used.

15.1.5. To know which precursors of fentanyl are controlled.

15.1.6. To understand the special toxicity of fentanyl and its derivatives as compared to other opiates.

15.2. Required Readings

15.2.1. CLIC Fentanyl Monograph, Version 1.0, 2009.

15.2.2. US Patent 3164600 1965, P. A. J. Janssen (FR1344366 may be substituted)

15.2.3. Hsu, F.-L. and Banks, H. D. “Fentanyl Synthetic Methodology: A Comparative Study” CRDEC 92-1332 (US Army)

15.2.4. Valdez, C. A., Leif, R. N, and Mayer, B.P. “An Efficient, Optimized Synthesis of Fentanyl and Related Analogs” PLoS One, 2014; 9(9): e108250 (from www.ncbi.nlm.nih.gov)

15.2.5. Siegfried, “Synthesis of Fentanyl” from Rhodium.WS https://www.erowid.org/archive/rhodium/chemistry/fentanyl.html

15.2.6. Fentanyl information and references available on the Keith Norman Disk.

15.3. Suggested Readings

15.3.1. Gupta, P. K. et al. “A Convenient One-Pot Synthesis of Fentanyl,” Journal of Chemical Research, July 2005 (pp. 452-453)

15.3.2. CLIC Meetings:

15.3.2.1. CLIC 2015 Fentanyl Trends, Recent Seizures, and Safe Analysis Practices**,** Danielle Farrell Drug Enforcement Administration-Western Laboratory

15.3.2.2. CLIC 2012 Case Studies of Two Fentanyl Synthesis Labs Seized in British Columbia, Canada, in 201, Jenny Shen, Drug Analysis Service Laboratory, Health Canada

15.4. Study/Discussion Exercises

15.4.1. Review the chemistry of piperidine and 4-piperidone and its importance to fentanyl synthesis.

15.4.2. Obtain MS of NNP, ANPP, and fentanyl. Which fragment peaks are conserved?

15.4.3. Propionyl chloride has been described as the 'smoking gun' of fentanyl labs. How would one analyzed this to ensure that it is not propionic acid or a related ester?

15.4.4. A reducing agent is necessary according to most published procedures. Is there a way of analyzing borohydride bases? What would be the appropriate wording for the report results?

15.4.5. Look up the LD50 values for fentanyl and compare them to the LD50 values of morphine, heroin, and methadone. What other special hazard is associated with fentanyl?

15.5. Practical Exercises

15.5.1. Draw out two synthetic approaches to the synthesis of fentanyl, and research the availability and price of materials using Fisher and/or online sources.

15.5.2. From your answers in the previous question, what types of chemical apparatus would be needed to carry out the synthesis? How would you describe these to scene responders?

15.5.3. Identify the common sources of structural change in analogs (e.g., acetylfentanyl, ring substitutions) and how the reagents change. What would be the expected changes in the MS of these changes?

16. YIELD ESTIMATES

16.1. **Objectives**

16.1.1. To develop an understanding of how to calculate a yield based upon the amount of precursors.

16.1.2. To develop an understanding of the inherent strength or weakness of estimates based on various criteria.

16.1.3. To be familiar with reporting requirements regarding “opinions and interpretations”.

16.2. **Required Readings**

16.2.1. Ely, R. A., “A Spreadsheet Program for the Determination of Volumes of One and Two Phase Liquids in Round Bottom Reaction Flasks,” Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 1, No. 3, July 1991, pp. 12-16.

16.3. **Suggested Readings**

16.3.1. Wojcik, C., “Safety Alert: New Cold Method Labs On Increase”, Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 2, No. 4, July 1992, pp. 2-3.

16.4. **Study/Discussion Exercises**

16.4.1. Explain what is meant by a yield calculation. What are some methods that can be used to calculate yields? What are some of the limitations of yield calculations?

16.4.2. If you have 100 tablets with 60 mg pseudoephedrine HCl per tablet, how much methamphetamine could theoretically be produced? What yield amount would you report and why?

16.4.3. Discuss why making yield estimates based on other variables such as the amount of red phosphorus, a reaction mixture or a tank of anhydrous ammonia are ill advised.

16.4.4. Discuss the differences between reporting yield within a lab report and testifying about yield in a courtroom. If you were asked to estimate the amount of methamphetamine that could be produced based on a particular synthesis route what would you say?

16.5. **Practical Exercises**

16.5.1. Use the latest version of the Drug Yield Calculator and the mass/volume of precursors and reagents from your previous methamphetamine synthesis to calculate the theoretical maximum yield and predicted amounts (range). How do these estimates compare with your actual yield? Print reports for these calculations.

16.5.2. Starting with one kilogram of saffrole, choose a method of MDMA synthesis and calculate the theoretical yield. Show your calculations for each step.

17. REPORT WRITING

17.1. **Objectives**

17.1.1. To become familiar with the requirements for report writing.

17.1.2. To develop confidence and skill in individual report writing skills.

17.2. **Required Readings**

17.2.1. Washington State Patrol Chemical Analysis Technical Procedures Manual section 2.10 and chapter 3.

17.3. **Suggested Readings**

17.3.1. Christian, Donnell R. Forensic Investigation of Clandestine Laboratories, Chapter 6 Opinions, CRC Press, 2004.

17.4. **Study/Discussion Exercises**

17.4.1. Review several completed clan lab cases performed by different analysts. Discuss these with your trainer.

17.4.2. Obtain and review example reports from several clan lab analysts throughout the division. Discuss these with your trainer.

17.5. **Guide**

17.5.1. Organize your report in a way to answer the questions posed by the submitter.

17.5.1.1. Was this a clandestine laboratory?

17.5.1.2. Were there controlled substances present?

17.5.1.3. What was being manufactured?

17.5.1.4. How much was being manufactured, or could be manufactured?

17.5.2. Connect items you identified to steps in the manufacture process, remember some items can be used in multiple steps.

17.5.3. If there is a scene report from the submitting agency, items from that could be used in forming your conclusion or included in your discussion, be sure to site the scene report.

17.5.4. Be aware of the legal definition of certain chemicals, and use appropriate terms in describing them in your report.

17.5.4.1. Legally anhydrous ammonia contains about 2% water, so it is not chemically anhydrous ammonia. Ammonia generated from ammonia salts will always contain water. Terms such as liquefied ammonia gas, or compressed ammonia gas should be used in the

report not liquid ammonia. This will distinguish the ammonia used in synthesis from the ammonia used in household cleaning.

17.5.5. Do not let your experience cause you to be overly specific in your conclusions in the report. Can the chemicals you identified be used in variations of the method you are used to seeing? If so, be more generic in your conclusion.

17.5.5.1. Do not call a synthesis method the red phosphorus method, unless you have red phosphorus identified. Calling it “a phosphorous and iodine method” will leave the option of phosphorus acids being used.

17.5.6. Is your report specific enough that a non-scientist can read it and understand why you reached your conclusions, but not so specific that it can be used as a recipe in a future clandestine laboratory?

17.5.6.1. Listing specific chemical sources could give future cooks too much information.

17.6. **Practical Exercises**

17.6.1. Obtain mock clan lab case notes from your trainer. These will include an item number, description of the item, and the analytical findings. Write a mock report based on all of the given information.

17.6.2. Discuss your mock clan lab reports with your trainer.

18. TESTIMONY

18.1. **Objectives**

18.1.1. Demonstrate a basic understanding of terms, legal decisions and issues relevant to clandestine laboratory cases.

18.1.2. Understand the differences between a controlled substances testimony and a clandestine laboratory testimony.

18.1.3. Understand how to prepare for a clandestine laboratory testimony.

18.1.4. Demonstrate how to effectively employ visual displays to aid in testimony.

18.1.5. Understand how to effectively testify in a clandestine laboratory case to connect the evidence analyzed with the description of the method used and the stages of manufacture.

18.2. **Required Readings**

18.2.1. The required reading in the Primary Foundation Training Manual, Law Basics and Court Testimony section.

18.2.2. Christian, Donn, “Courtroom Presentation of Clandestine Drug Laboratory Cases” Journal of the Clandestine Laboratory Investigating Chemists Association, Vol. 2, No. 4, October 1992, pp. 20-24.

18.2.3. Courtney, M., “In Search of Reason: Evaluating Clandestine Labs for Court” Journal of the Clandestine Laboratory Investigating Chemists, Vol. 2, No. 4, 1992, pp. 29-32.

18.3. **Suggested Readings**

18.3.1. Christian, Donnell R. Forensic Investigation of Clandestine Laboratories, Chapter 7 Testimony, CRC Press, 2004.

18.4. **Study/Discussion Exercises**

18.4.1. Your instructor will give you a clandestine laboratory case file they wrote. You will read the case file and prepare for court as if you are testifying to the file, and be prepared to discuss how the case was analyzed and what conclusions could be drawn from the case.

18.4.2. Review any transcripts of clandestine laboratory testimony and discuss the scientists approach to the testimony with your instructor.

18.4.3. Describe and explain aloud the various methods of manufacture as you would present them to a jury.

18.5. **Practical Exercises**

18.5.1. Conduct an analysis on a sample provided to you by your instructor and write a report on the results of this analysis. Participate in a mock court trial covering the examination and your report.

18.5.2. Observe clandestine laboratory court testimony by other members of the laboratory staff.

19. COMPETENCY EXAMINATION

19.1. **Objectives**

19.1.1. To instill self-confidence in the analytical ability of the student.

19.1.2. To ensure that the student has acquired the specialized knowledge, skills and abilities in the discipline of clandestine laboratory examinations.

19.1.3. To ensure appropriate documentation and report writing skills.

19.1.4. To ensure appropriate techniques and confidence for court presentation.

19.2. **Study/Discussion Exercises**

19.2.1. Discuss the results of the competency evaluation (see practical exercises below) with the instructor.

19.2.2. Evaluate the training plan and provide recommendations for future revisions.

19.3. **Practical Exercises**

19.3.1. Complete a written or oral examination.

19.3.2. Work at least two mock cases and write reports.

19.3.3. Ask clan lab analysts in your lab about their court experiences. What are some of the questions they typically receive as witnesses and how do they answer them? What are some lessons they have learned about expert testimony?

19.3.4. Observe court testimony of other clan lab analysts (if possible).

19.3.5. Undergo a mock trial. (Optional for experienced analysts with extensive courtroom experience.)

20. APPENDIX A: IODINE

20.1. **Purpose**

20.1.1. This document outlines the general methods of analysis used to examine evidence for the presence of iodine or iodide.

20.2. **References**

20.2.1. O'Neil, Maryadele, editor, Merck Index, 13th edition, 2001, Merck & Co., Inc.

20.2.2. Daintith, John, editor, Dictionary of Chemistry, 1981 Barnes & Noble Books.

20.2.3. Bogert, L. Jean, Fundamentals of Chemistry, 1941, W.B. Saunders Company.

20.2.4. McKibben, Tim, Analyses of Inorganic Components Found in Clandestine Drug Laboratory Evidence, CLIC Journal, October 1995.

20.3. **Scope**

20.3.1. The methods described in this section are suitable for the analysis of samples containing or suspected to contain iodine or iodide.

20.3.2. Due to the variability of received samples, no part of this method should be construed to be exclusive or absolute. Alternative methods of analysis are acceptable with proper attention to quality assurance issues, such as the run of a positive and negative control where appropriate. The exact analytical protocol used will be based on the type of sample and the training and experience of the analyst.

20.4. **Minimum Identification Criteria for Elements and Inorganic Compounds**

20.4.1. Iodine is an essential chemical as defined in the Clandestine Laboratory Analysis chapter of the Materials Analysis Technical Procedures.

20.5. **Methods**

20.5.1. Starch/color test

20.5.1.1. "One of the most characteristic properties of starch is that it gives a blue compound with iodine." (Reference 20.2.3, Bogert, p326).

20.5.1.2. A small amount of sample is dissolved in water and then either powdered starch or an aliquot of aqueous (approximately 1%) starch solution is added. A positive test for elemental iodine is the formation of a blue-black color. If the sample is aqueous, it needs to be made acidic and an oxidizer such as nitric acid or hydrogen peroxide should be added, then the liquid can be added to powdered starch ot to an aqueous starch solution.

20.5.2. Solubility/color test

20.5.2.1. Place a small amount of sample in a test tube (add MeOH if sample is a solid) and add 1-2mL of hexane. A purple color will develop if iodine is present. If iodide is present, the addition of nitric acid (HNO3) to the test tube will oxidize the iodide to elemental iodine and a purple color will appear in the hexane layer. The hexane layer may then be used for GC/MS analysis . This test may also be done with chloroform or pentane replacing the hexane.

20.5.2.2. Elemental iodine added to a test tube with layers of chloroform/hexane and methanol will produce purple in the non-polar layer and brown in the alcohol layer. (Reference 20.2.1, Merck)

20.5.3. XRF/SEM-EDX

20.5.3.1. Put a small amount of sample in an appropriate container. Or, add AgNO3 to an aqueous extract of your sample to form the yellow AgI precipitate. This precipitate can then be collected, dried, and run on the XRF. Liquid samples may be spotted onto filter paper, dried, and examined as above. In these situations, it is recommended to compare the sample data to “filter paper background” data. Note: XRF data cannot distinguish between iodine and iodide. The lab should possess an archived copy or file of the relevant data run with lab standards.

20.5.4. GC/MS of iodine

20.5.4.1. A solution of elemental iodine in chloroform or hexane may be run on the GC/MS using a low-temperature ramp. Make a solution of 1 prill or flake of elemental iodine in approximately 1 mL of chloroform or hexane. Iodide-containing samples can be oxidized with hydrogen peroxide or nitric acid and the resulting iodine transferred to a chloroform, pentane, or hexane layer for analysis by GC/MS, this is best done from acidic solutions.

20.5.5. Raman

20.5.5.1. Place the sample in an appropriate container, to prevent sublimation, to analyze. Either use a low laser power setting, an appropriate filter, or mix a small amount of the sample with potassium bromide.

20.5.6. CE

20.5.6.1. Take a small amount of the sample, extract with CE grade water and analyze using the CE anion method (JFS 2006, Vol. 51(1) pp 82-86. Alternate columns may be used.

20.5.7. Analysis of iodine tincture or tincture waste

20.5.7.1. Iodine can be precipitated or extracted from tincture of iodine by a combination of hydrogen peroxide and hydrochloric acid or household bleach and sulfuric acid. If your sample is tincture waste, it will likely be acidic (test diluted sample with pH strip). Precipitation tests on adulterated tincture waste usually are consistent with HCl rather than HI. Tests 1-6 may be performed on precipitated or extracted iodine.

21. APPENDIX B: RED PHOSPHORUS

21.1. **Purpose**

21.1.1. This document outlines the general methods of analysis used to examine evidence for the presence of phosphorus or hypophosphorous acid.

21.2. **References**

21.2.1. O'Neil, Maryadele, editor, Merck Index, 13th edition, 2001, Merck & Co., Inc.

21.2.2. Daintith, John, editor, Dictionary of Chemistry, 1981 Barnes & Noble Books.

21.2.3. Bogert, L. Jean, Fundamentals of Chemistry, 1941, W.B. Saunders Company.

21.2.4. McKibben, Tim, Analyses of Inorganic Components Found in Clandestine Drug Laboratory Evidence, CLIC Journal, October 1995.

21.2.5. Kansas Bureau of Investigation, Phosphorus Derivation Technique, KBI.

21.2.6. Chamot, E. and Mason, C, Handbook of Chemical Microscopy: Volume II, 1931, John Wiley & Sons, Inc.

21.2.7. Schieferecke, J, Red Phosophorus Analysis Using a Gas Chromatograph / Mass Spectrometer, CLIC Journal, July 2000.

21.3. **Scope**

21.3.1. The methods described in this section are suitable for the analysis of samples containing or suspected to contain red phosphorus, phosphorus of another allotropic form (white or black).

21.3.2. Due to the variability of received samples, no part of this method should be construed to be exclusive or absolute. Alternative methods of analysis are acceptable with proper attention to quality assurance issues, such as the run of a positive and negative control where appropriate. The exact analytical protocol used will be based on the type of sample and the training and experience of the analyst.

21.4. **Minimum Identification Criteria**

21.4.1. Phophorus is an essential chemical as defined by chapter three of the CATP. To report red phosphorus the sample must be red or purple in color and give a positive ignition test, otherwise you must report, phosphorus, phosphorus salts, phosphorus acids, or the specific acid identified.

21.5. **Methods**

21.5.1. Ignition test

21.5.1.1. When red phosphorus is heated in air, it turns to white phosphorus and then reacts with oxygen to form a white solid P2O5. For the flame test, pack a disposable glass pipette

with an aliquot of sample (preferably dry) between glass wool plugs (or simply place the powder in a glass pipette). Put a squeezable bulb on the pipette and hold the pipette over an alcohol burner to heat the sample. When the temperature is high enough, the red phosphorus will convert to white/yellow phosphorus and when air is forced through will ignite, giving a pale green-to-white flame. When air is blown in and out of the pipette using the bulb, you may observe the pale vortex of flame inside the pipette.

21.5.1.2. The white smoke coming out of the pipette should be blown into a test tube containing water. The water should be strongly acidic due to the formation of phosphoric acid from P2O5 reacting with water (References 21.2.2, Daintith; 21.2.3, Bogert; and 21.2.4, McKibben)

21.5.2. XRF/SEM

21.5.2.1. Put a small amount of dry powder or sample cutting with powder residue on a plastic slide with double-sticky tape. Cover with mylar film and run on the XRF instrument. It may also be analyzed with any other appropriate container for XRF. Moist samples will also work, but with poorer emission levels. If the red phosphorus has been used, trace iodide is often present. Elements such as Si, Al, Ca, Fe and Ti indicate that the phosphorus may have come from matchbook striker plates. The lab should possess an archived copy or file of the relevant data run with lab standards. Note: XRF data cannot distinguish between phosphorus and phosphates or other anions containing phosphorus.

21.5.2.2. Note: If an SEM is used, small particles of glass can often be seen and identified if the red phosphorus came from matchbooks.

21.5.3. GC/MS

21.5.3.1. Place approximately 3 mg sample in a test tube. Purge the sample with dry nitrogen. Hold the test tube over the Bunsen burner flame; remove when you see smoke inside. Ready a pipette with approximately 2 mL chloroform. Quickly wash chloroform down the sides of the test tube. Inject sample into your GC/MS system. The GC/MS should use a temperature ramp of approximately 35 ºC to 200 ºC at 35 ºC per minute. (References 21.2.7, Schieferecke)

21.5.3.2. Note: Another method for this procedure is to blow the smoke from the ignition test (21.5.1) directly into the chloroform, and then proceed with the GC/MS analysis as above.

21.5.4. Raman

21.5.4.1. The sample may be run neat on the Raman, using appropriate Raman accessories. A sample that has been used to synthesize methamphetamine may have to be cleaned before analysis.

21.5.4.2. Safety Note: Red Phosphorus can ignite with a strong heat source, use a low laser setting, and if needed a filter, or mix the red phosphors with potassium bromide.

21.5.5. CE

21.5.5.1. Take a small amount of the sample, extract with CE grade water and analyze using the CE anion method (JFS 2006, Vol. 51(1) pp 82-86). Alternate columns may be used.

21.5.5.2. See also: Person, E.C., Knops, L.A., Heegel, R.A., and Northrop, D.M., "Phosphorus-Containing Reducing Agents: A review of their chemistry and use in the manufacture of methamphetamine and the significance of observed phosphate, phosphite, and hypophosphite in clandestine laboratory casework.", J. of Clandestine Laboratory Investigating Chemists' Association, Vol. 18, No. 2, April 2008, pp. 7-44.

22. APPENDIX C: LITHIUM

22.1. **Purpose and Scope**

22.1.1. This appendix outlines the general methods of analysis used to examine evidence for the presence of lithium metal or lithium salts. Alternative methods of analysis are acceptable with proper attention to quality assurance issues, such as the run of a positive and negative control where appropriate. The exact analytical protocol used will be based on the type of sample and the training and experience of the analyst.

22.2. **References**

22.2.1. O'Neil, Maryadele, editor, Merck Index, 14th edition, 2006, Merck & Co., Inc.

22.2.2. McKibben, Tim, Analyses of Inorganic Components Found in Clandestine Drug Laboratory Evidence, CLIC Journal, October 1995.

22.2.3. Anderson, O. Carl, Lithium Spot Test, CLIC Journal, July 2000.

22.2.4. Worley, Dwain, Lithium Analysis, CLIC Fall Meeting 2004.

22.2.5. DalCason, Terry, The Birch/Benkeser Reactions and Potential Modifications in the Manufacture of "Nazi Dope", CLIC Fall Meeting 2001.

22.2.6. Agilent Technologies, Cation Solutions Kit Manual, PN 5064-8206.

22.2.7. Chamot, E. and Mason, C, Handbook of Chemical Microscopy: Volume II, 1989, McCrone Research Institute., p. 77.

22.3. **Analytical Information**

22.3.1. Lithium metal and lithium salts are considered essential chemicals as defined by the Clandestine Laboratory Analysis chapter of the MATP.

22.4. **Methods**

22.4.1. Reactivity / pH test

22.4.1.1. Lithium metal is water-reactive with production of LiOH, H2, and heat. A tiny fragment of suspected lithium metal may be placed in DI water to observe any reactivity. The pH of the aqueous solution can be subsequently tested (1.0N solution of LiOH has a pH of 14). This is only a presumptive test for lithium as other alkali and alkaline earth metals are also water-reactive with the formation of hydroxides.

22.4.2. XRF/SEM-EDX

22.4.2.1. Non-instrumental for lithium

22.4.2.2. Lithium is too light to emit XRF radiation. However, you can screen your sample for the presence of calcium or strontium, which give flame test results that may be mistaken for

lithium. Sample preparation should follow appropriate technical procedures. Lithium waste should be dried to allow any ammonia vapors to dissipate prior to analysis.

22.4.3. Flame test

22.4.3.1. Lithium metal and lithium salts exhibit a characteristic crimson color (670.8 nm) in flame. (Reference 22.2.1, Merck) Both calcium metal (red-orange) and strontium metal (brilliant red) may produce similar colors. However, both calcium and strontium can be detected by XRF, SEM/EDX, and all three may be detected by CE. Sodium, which gives a bright yellow-orange flame, may interfere with the crimson of lithium. Dydimium safety glasses or similar glasses that reduce sodium emissions may be helpful to observe the presence of lithium in the presence of sodium.

22.4.4. Precipitation test for lithium

22.4.4.1. This test (Reference 22.2.3, Anderson) uses an iron (III) periodate/potassium hydroxide solution (combined with saturated NaCl for greater sensitivity) to form a precipitate indicative of lithium. Negative and positive controls must be run concurrently. Alkaline earth metals may give false positives. The test may be modified (steps 1-4) to improve specificity. (Reference 22.2.4, Worley) The modified precipitation test for lithium incorporates a pre-step that will precipitate out calcium and strontium salts (using ammonium phosphate) prior to the addition of the iron (III) periodate reagent. This test is much more specific for lithium cations. It is recommended that the analyst use the modified version of the precipitation test in combination with a red flame test, XRF, or SEM/EDX to report lithium metal or lithium salts.

22.4.4.2. Reagents

22.4.4.2.1. Note: These keep indefinitely when stored under refrigeration.

22.4.4.2.2. Saturated NaCl solution

22.4.4.2.3. Iron (III) Periodate solution

- 2 grams of potassium periodate (KIO4) in 10mL 2N KOH
- Mix 37 mL H2O with 3mL of a 10% FeCl3 solution
- Add FeCl3 solution to iron periodate solution, dilute up to 100mL with 2N KOH

22.4.4.2.4. Ammonium hydroxide solution

22.4.4.2.5. 10% (by weight) aqueous ammonium phosphate solution

22.4.4.3. Procedure

22.4.4.3.1. Analyze your sample concurrently with a deionized water blank and a positive lithium solution.

22.4.4.3.2. Using ten-drop aliquots of your sample solution, blank, and positive control, basify each to pH 11 or greater with aqueous ammonium hydroxide.

22.4.4.3.3. Add 2 drops ammonium phosphate solution. Calcium and strontium will form a precipitate; lithium will remain in solution.

22.4.4.3.4. Spin down and transfer the clear supernatants to clean test tubes.

22.4.4.3.5. To each, add 2 drops of saturated NaCl solution, 2 drops of your test solution (centrifuged/transferred supernatant from step 4) and 4 drops iron (III) periodate solution.

22.4.4.3.6. A positive reaction for lithium cations is an off-white precipitate. Be sure to compare to the blank, as even the blank may have slight turbidity. Make your observations immediately; cloudiness may form in all the tubes if allowed to sit. If there are no immediately conclusive results, the test tubes may be run under hot water for 30 seconds and re-observed.

22.4.4.4. FTIR

22.4.4.4.1. Instrumental, structural data

22.4.4.4.2. Lithium salts, such as lithium hydroxide and lithium carbonate, can be analyzed using FTIR and compared to a laboratory or reference standard. Be aware that lithium waste may have impurities and lithium hydroxide will absorb carbon dioxide and water from air to form lithium carbonate. Direct analysis of such solid waste may not provide a pure spectrum. Lithium waste may be dissolved in water and washed with organic solvents to remove impurities. The aqueous phase can be subsequently dried to obtain a spectrum of lithium carbonate.

22.4.4.4.3. Refer to the FTIR Technical Procedures for further information on using this instrument in casework.

22.4.5. Raman

22.4.5.1. Instrumental, structural data

22.4.5.2. Similar to FTIR, lithium salts may be analyzed on a Raman instrument and compared to a laboratory or reference standard. Refer to the Raman Technical Procedures for further information on using this instrument in casework.

22.4.6. CE

22.4.6.1. Instrumental

22.4.6.2. Cations (e.g. NH4+, Na+, Ca+2, Li+) can be analyzed using the method provided by Agilent® (Reference 22.2.6). While cations will not provide UV data, good cation peak separation is achieved. A cation standard should be run the same day as any sample. Additionally, it is not uncommon for peak drift to occur with this method. Therefore, using an internal standard or spiking a second sample with an appropriate chemical to show co-elution should be performed. Additional information regarding CE use is detailed in the capillary electrophoresis chapter of the MATP.

22.4.7. Sodium or Ammonium Carbonate Crystal Test

22.4.7.1. Add several small pieces of sodium carbonate to a microscope slide containing a concentrated sample solution. Viewed under a polarizing light microscope, hexagonal stars or plates are positive results for lithium.

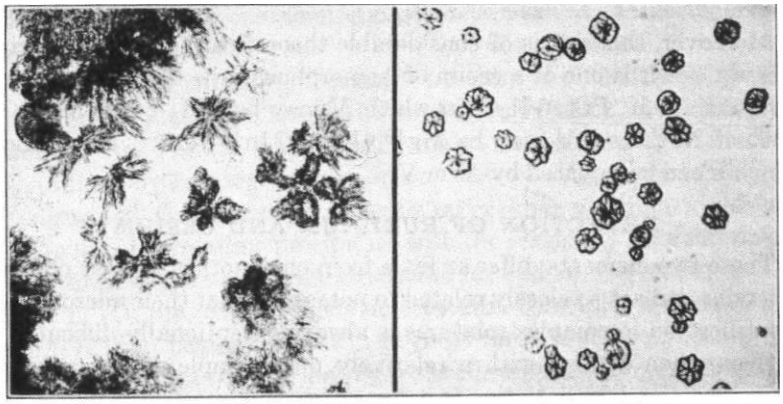


FIG. 50. Lithium with Ammonium Carbonate. 100×.

FIG. 51. Lithium with Ammonium Carbonate. 200×.

Figure 1: photo from reference 22.2.7

23. APPENDIX D: AMMONIA

23.1. **Purpose and Scope**

23.1.1. This appendix outlines the general methods of analysis used to examine evidence for the presence of ammonia or ammonium salts. Alternative methods of analysis are acceptable with proper attention to quality assurance issues, such as the run of a positive and negative control where appropriate. The exact analytical protocol used will be based on the type of sample and the training and experience of the analyst.

23.2. **References**

23.2.1. O'Neil, Maryadele, editor, Merck Index, 14th edition, 2006, Merck & Co., Inc.

23.2.2. DalCason, Terry, The Birch/Benkeser Reactions and Potential Modifications in the Manufacture of "Nazi" Dope, CLIC Fall Meeting 2001.

23.2.3. Worley, Dwain, Evaluation of Ammonium Test Paper, CLIC Journal, April 2002.

23.2.4. Dawson, Nick, The Sodium-Ammonia "Nazi" Method of Methamphetamine Synthesis: A Historical Overview, Methodology and Case Review, CLIC Journal, July 1995.

23.2.5. Chamot, E. and Mason, C, Handbook of Chemical Microscopy: Volume II, 1940, John Wiley & Sons, Inc., p. 65.

23.2.6. Smiley, J.C., et al., Analysis of Anhydrous Ammonia Via the Precipitation of Ammonium Salt, CLIC Journal, January 2001.

23.2.7. Agilent Technologies, Cation Solutions Kit Manual, PN 5064-8206.

23.3. **Analytical and Legal Information**

23.3.1. Ammonia and ammonium salts are considered essential chemicals as defined by the Clandestine Laboratory Analysis chapter of the MATP.

23.3.2. It is a class B felony to possess pressurized ammonia gas or pressurized ammonia gas solution with intent to manufacture methamphetamine (RCW 69.50.440).

23.4. **Methods**

23.4.1. pH test

23.4.1.1. Ammonia in water has a pH between 10 and 12. Also a wet pH strip can be inserted under the lid of a sample container to test the headspace pH before the sample is further examined. The headspace pH should also be approximately 10-12.

23.4.2. Vapor phase FTIR of Ammonia

23.4.2.1. Instrumental, structural data

23.4.2.2. Ammonia vapor has a characteristic FTIR pattern with absorbances in the following regions: 3550 – 3100 cm-1, 1800 – 1450 cm-1 (overlaps with water vapor), and 1250 – 600 cm-1. A standard comparison can be made to the ammonia vapors from a laboratory grade bottle of ammonium hydroxide. Ammonia vapors should be collected at half a wavenumber or less resolution.

23.4.2.3. Collect the headspace vapors above a sample, transfer to a vapor cell, and collect a spectrum. If necessary, the addition of a sodium hydroxide solution to a sample may be sufficient to force enough ammonia vapor from the sample to collect a satisfactory spectrum. The air above an analyst's workbench should be used as a background spectrum at the time of analysis.

23.4.3. Ammonium test strips

23.4.3.1. Ammonium test strips are available from JT Baker®, and other manufacturers. These utilize a strip impregnated with mercury potassium iodide and a separate 28% sodium hydroxide solution. The test strip will change color in a range to indicate the presence of ammonium between 10 and 400 mg/L and is very specific for the presence of ammonium (Reference 23.2.3, Worley). The lot number of the testrips and sodium hydroxide solution must be recorded. The used strips should be discarded with care due to the mercury indicating compound.

23.4.4. Microchemcial test for Ammonia or Ammonium salts

23.4.4.1. For ammonia vapors, perform using the hanging drop method by placing a drop of 5% chloroplatinic acid (H2PtCl6) on a slide that has been inverted over the sample. For solid samples, a drop of 3N NaOH may be used to encourage volatilization of the ammonia vapor from an ammonium salt.The resulting crystals are small yellow doublet pyramids, which usually rest on one face to produce what looks like small hexagons. The test may be performed directly on solid samples; however, similar crystals are formed by potassium salts with H2PtCl6. Therefore, solid samples should be treated with NaOH to test for ammonia vapors. Potassium ions do not volatilize. (Reference 23.2.5, Chamot and Mason)

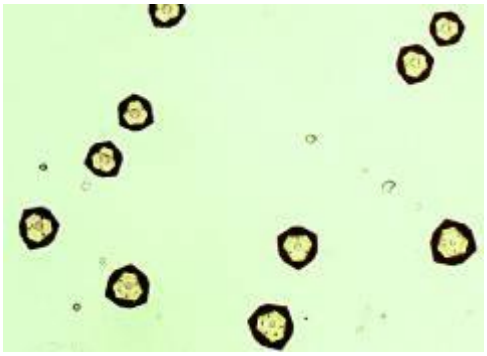


Figure 1: Photo from Reference 23.2.5

23.4.5. FTIR

23.4.5.1. Instrumental, structural data

23.4.5.2. Solid ammonium salts can be run on an IR and compared to a laboratory or reference standard. Refer to the FTIR Technical Procedures for further information on using this instrument in casework.

23.4.5.3. Ammonia vapors may also be converted to an ammonium salt, dried, and collected for FTIR. Use either a hanging drop of concentrated HCl or collect ammonia vapors in a pipette and blow into or across a drop of concentrated HCl to form ammonium chloride. Acetone may be used to precipitate the salt formed. (Reference 23.2.6, Smiley, et. al.)

23.4.6. Raman

23.4.6.1. Instrumental, structural data

23.4.6.2. Ammonium salts may be analyzed on a Raman instrument and compared to a laboratory or reference standard. Refer to the Raman chapter of the MATP for further information on using this instrument in casework.

23.4.6.3. Similar to FTIR, ammonia vapors may also be converted to an ammonium salt, dried, and collected for analysis by Raman.

23.4.7. CE

23.4.7.1. Instrumental

23.4.7.2. Cations (NH_4^+, K^+, Na^+, Ca^{+2}, Mg^{+2}, Li^+) can be analyzed using the method provided by Agilent® (Reference 23.2.7). While cations will not provide UV data, good cation peak separation is achieved. A cation standard should be run the same day as any sample. Additionally, it is not uncommon for peak drift to occur with this method. Therefore, using an internal standard or spiking a second sample with an appropriate chemical to show co-elution should be performed. Additional information regarding CE use is detailed in the capillary electrophoresis chapter of the MATP.

24. APPENDIX E: ACIDS

24.1. Purpose and Scope

24.1.1. This appendix outlines the general methods of analysis used to examine evidence for the presence of acids commonly encountered in clandestine laboratory analysis. Alternative methods of analysis are acceptable with proper attention to quality assurance issues, such as the run of a positive and negative control where appropriate. The exact analytical protocol used will be based on the type of sample and the training and experience of the analyst.

24.2. References

24.2.1. O'Neil, Maryadele, editor, Merck Index, 13th edition, 2001, Merck & Co., Inc.

24.2.2. McKibben, Tim, Analyses of Inorganic Components Found in Clandestine Drug Laboratory Evidence, CLIC Journal, October 1995.

24.2.3. Oulton, S and Skinner, H., Identification of Common Inorganic Acids Encountered at Clandestine Laboratories, CLIC Journal, October 1998.

24.2.4. Skinner, H. and Oulton, S., Identification and Quantitation of Hydriodic Acid Manufactured from Iodine, Red Phosphorus and Water, CLIC Journal, October 1995.

24.2.5. Chamot, E. and Mason, C, Handbook of Chemical Microscopy: Volume II, 1931, John Wiley & Sons, Inc.

24.2.6. Knops, L., Northrop, D., and Person, E., Capillary Electrophoretic Analysis of Phosphorus Species in Clandestine Methamphetamine Laboratory Samples, Journal of Forensic Science, 51(1), 2006.

24.3. Analytical Information

24.3.1. Hydriodic acid and hypophosphorus acid are essential chemicals as defined by the Clandestine Laboratory Analysis chapter of the MATP. Other acids, such as sulfuric acid and hydrochloric acid, will be regarded as supplementary chemicals as defined by the Clandestine Laboratory Analysis chapter of the MATP.

24.4. General Methods for distinguishing common inorganic acids

24.4.1. Capillary Electrophoresis (CE)

24.4.1.1. Many anions can be analyzed using the method provided by Knops (Reference 24.2.6). An anion standard should be run the same day as any sample.

24.4.1.2. Refer to the Technical Procedures for further information on using CE in casework.

24.4.2. Precipitation Tests

24.4.2.1. The precipitates can be further analyzed using XRF or SEM/EDX.

Table 1: Table from Reference 24.2.3, Oulton

Acid	Silver Nitrate	Ammonium Hydroxide	Barium Nitrate	Basified/ Barium Nitrate
Hydriodic	yellow precipitate	white precipitate	no precipitate	no precipitate
Hydrobromic	pale yellow precipitate	dissolves	no precipitate	no precipitate
Hydrochloric	white precipitate	dissolves	no precipitate	no precipitate
Hypophosphorus	black precipitate	no change	no precipitate	no precipitate
Sulfuric	white precipitate	dissolves	white precipitate	white precipitate
Phosphoric	yellow precipitate	dissolves	no precipitate	white precipitate
Nitric	no precipitate	N/A	no precipitate	no precipitate
Phosphorus	no precipitate	N/A	no precipitate	no precipitate

24.4.2.2. Silver Nitrate

- 1 to 2 drops of a 5% AgNO3 solution is added to the sample. If a precipitate is formed, add concentrated ammonium hydroxide and observe if the precipitate dissolves. Compare the results to the table above.

24.4.2.3. Barium Nitrate

- 1 to 2 drops of a 5% Barium nitrate solution is added to the sample. Observe the formation of a precipitate, if any. Compare the results to the table above.

24.4.2.4. Basic Barium Nitrate

- 1 to 2 drops of a 50% sodium hydroxide solution are added to the sample. Add enough sodium hydroxide to ensure the solution is basic. Add 1 to 2 drops of 5% barium nitrate solution to the sample. Observe a precipitate, if any. Compare the results to the table above.

24.4.2.5. Ammonium Salt

- 0.5 milliliters of concentrated ammonium hydroxide is added to a 50 milliliter beaker. One to two drops of the acid are slowly added to the ammonium hydroxide. The pH should be greater than 8. Add approximately 40 milliliters of acetone to the beaker. This will precipitate the ammonium salt. Filter and dry the precipitate for analysis by FTIR or Raman. Data should be compared to a positive control.

24.4.3. Additional Methods for Hydrochloric Acid (HCl)

24.4.3.1. pH

24.4.3.1.1. Sufficiently concentrated HCl solutions have a pH of 0-1. A pH test strip moistened with water and held over the solution. will have a vapor pH of 0-1 for HCl and nitric acid; whereas, sulfuric acid, hydriodic acid, hypophosphorus acid, and phosphoric acid will not have an acidic vapor.

24.4.3.2. FTIR

24.4.3.2.1. HCl gives a unique FTIR vapor phase spectrum, showing two sets of evenly-spaced absorbances centering around 2900 wavenumbers. If this test is performed on an HCl sample and no discrepancies are noted in any other tests, HCl may be reported without the "consistent with …" phrase. The lab should possess an archived copy or file of the spectrum run with a lab standard or a literature reference.

24.4.4. Additional Methods for Sulfuric Acid H2SO4

24.4.4.1. Microcrystalline test

24.4.4.1.1. A positive test for sulfates with silver nitrate to form crystals of silver sulfate is a good indicator of sulfuric acid if the pH is strongly acidic. On a slide, mix one drop of a dilute sample solution to one drop of aqueous silver nitrate. The crystal formation is rapid, appearing as flat diamonds which are birefringent and continue to grow until they exhibit an "internal skeleton". (Reference 24.2.5, Chamot and Mason)

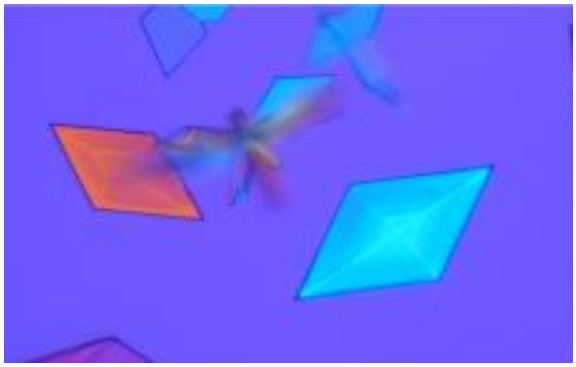


Figure 1: Photo Courtesy of Oregon State Police Crime Laboratory

25. APPENDIX F: SODIUM

25.1. Purpose and Scope

25.1.1. This appendix outlines the general methods of analysis used to examine evidence for the presence of sodium metal or sodium salts. Alternative methods of analysis are acceptable with proper attention to quality assurance issues, such as the run of a positive and negative control where appropriate. The exact analytical protocol used will be based on the type of sample and the training and experience of the analyst.

25.2. References

25.2.1. O'Neil, Maryadele, editor, Merck Index, 13th edition, 2001, Merck & Co., Inc.

25.2.2. McKibben, Tim, Analyses of Inorganic Components Found in Clandestine Drug Laboratory Evidence, CLIC Journal, October 1995.

25.2.3. Chamot, E. and Mason, C, Handbook of Chemical Microscopy: Volume II, 1931, John Wiley & Sons, Inc.

25.2.4. Agilent Technologies, Cation Solutions Kit Manual, PN 5064-8206.

25.3. Analytical Information

25.3.1. Sodium may be encountered in a clandestine lab situation as part of NaOH, a strong base, or, rarely, as sodium metal. Sodium metal can be produced clandestinely by electroplating sodium metal onto a cathode (typically iron) by running an electric current through molten sodium hydroxide. Sodium and sodium salts are considered essential chemicals as defined by the Clandestine Laboratory Analysis chapter of the MATP.

25.4. Methods

25.4.1. Reactivity / pH Test

25.4.1.1. Sodium metal will react with water to produce NaOH, H2, and heat. If the item is thought to be sodium metal, a very small piece may be added to deionized water to observe any reactivity. Aqueous solutions of sodium metal and sodium hydroxide produce a pH of 13-14 and are exothermic. Because other alkali and alkaline earth metals also react with water, this is only a presumptive test.

25.4.2. XRF or SEM-EDX

25.4.2.1. If using XRF, put a small amount of sample on a plastic slide with double-sticky tape. Sodium is usually too light to be detected using XRF, therefore, the sample may be run without the normal mylar covering and for a longer period of time.

25.4.2.2. Sample preparation should follow appropriate technical procedures. Refer to the Technical Procedures for further information on using these instruments in casework.

25.4.3. Capillary Electrophoresis (CE)

25.4.3.1. Cations (NH4+, K+, Na+, Ca+2, Mg+2, Li+) can be analyzed using the method provided by Agilent® (Reference 25.2.4). While cations will not provide UV data, good cation peak separation is achieved. A cation standard should be run the same day as any sample. Additionally, it is not uncommon for peak drift to occur with this method. Therefore, using an internal standard or spiking a second sample with an appropriate chemical to show co-elution should be performed.

25.4.3.2. Refer to the Technical Procedures for further information on using CE in casework.

25.4.4. Raman

25.4.4.1. Instrumental, structural data

25.4.4.2. Sodium salts may be analyzed on a Raman instrument and compared to a laboratory or reference standard. Refer to the Raman Technical Procedures for further information on using this instrument in casework.

25.4.5. Precipitation tests for hydroxide or chloride

25.4.5.1. Add an aqueous silver nitrate solution to the sample. If present, the hydroxide anion will react to form a brown (AgOH) precipitate.

25.4.5.2. If NaCl, is suspected, add an aqueous silver nitrate solution to the sample. Chlorine anions will form a thick white (AgCl) precipitate that will gradually disappear with the further addition of ammonium hydroxide. Be sure the original sample solution is neutral to rule out the presence of HCl in the sample.

25.4.6. Flame test

25.4.6.1. An aqueous solution on a platinum wire gives a yellow color (589.2 nm) in flame. (Reference 25.2.1, Merck). Sodium may interfere with other cations, such as lithium, in a flame test.

26. APPENDIX G: GENERAL ANALYTICAL GUIDELINES AND APPROACHES

Technical Suitability	Organic controlled substances	Organic precursors and by-products	Carbohydrates, Fats/oils, Amino Acids	Solvents	Inorganic elements	Inorganic neutral compounds	Inorganic salts	Inorganic ionic bases	Inorganic aqueous acids	Inorganic Oxidizers	Inorganic Reducers	Water
GC/MS												
GC/IR												
Raman												
IR												
SEM/EDX												
XRF												
CE												
HPLC												
TLC												
Microcrystalline tests												
Chemical wet tests												
Spectroscope												
Pharmaceutical Identification												

Key

generally useful

limited or requiring derivatization

not recommended except under special circumstances

Table 1: Suggested Analytical Techniques for Classes of Materials Encountered in Clan Lab Analysis

This table relates the general suitability of a given analytical technique to the different types of chemical substances (by class) that have been encountered in clan lab evidence. The green color indicates that the technique is generally very useful for analysis of that class of compound, and that methods exist within the system that are ready to use. Yellow indicates that the technique has some limitations – these limitations include (but are not limited to) small subsets of examples within the class that are amenable to the technique; extensive preparatory work to use the technique; in-house methods not readily available that would require verification or validation. Red indicates that the technique is not generally considered useful for the identification of this class of substances, and others should be chosen.

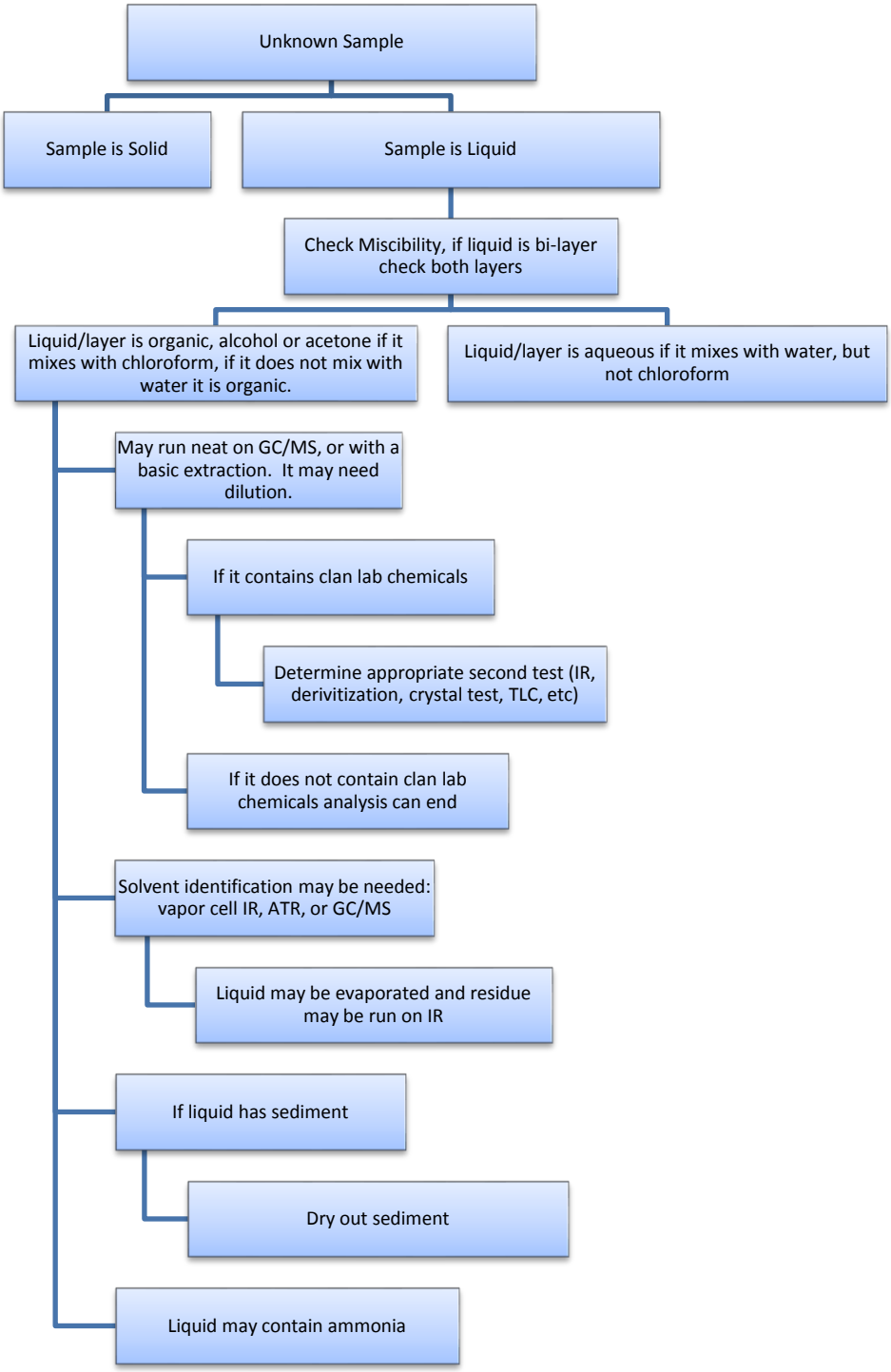
Associations	Confirmatory ID	Consistent With	Indications Of
GC/MS	With in-house reference material for Rt and MS match	With approved MS match	Incomplete MS
GC/IR	With in-house reference material for Rt and IR match	With approved IR match	???
Raman	With approved reference spectrum	Weak spectrum or presence of impurities	mixtures
IR	With approved reference spectrum	Weak spectrum or presence of impurities	mixtures
SEM/EDX		With approved reference spectrum	mixtures with bands of similar energy
XRF		With approved reference spectrum	mixtures with bands of similar energy
CE	n/a	by comparison to in-house reference material	
HPLC	n/a	by comparison to in-house reference material	
TLC	n/a	by comparison to in-house reference material	
Microcrystalline tests	n/a	by comparison to in-house reference material	
Chemical wet tests	n/a	by comparison to in-house reference material	
Spectroscope	n/a	by comparison to in-house reference material	
Pharmaceutical Identification	n/a	by comparison to in-house reference source	

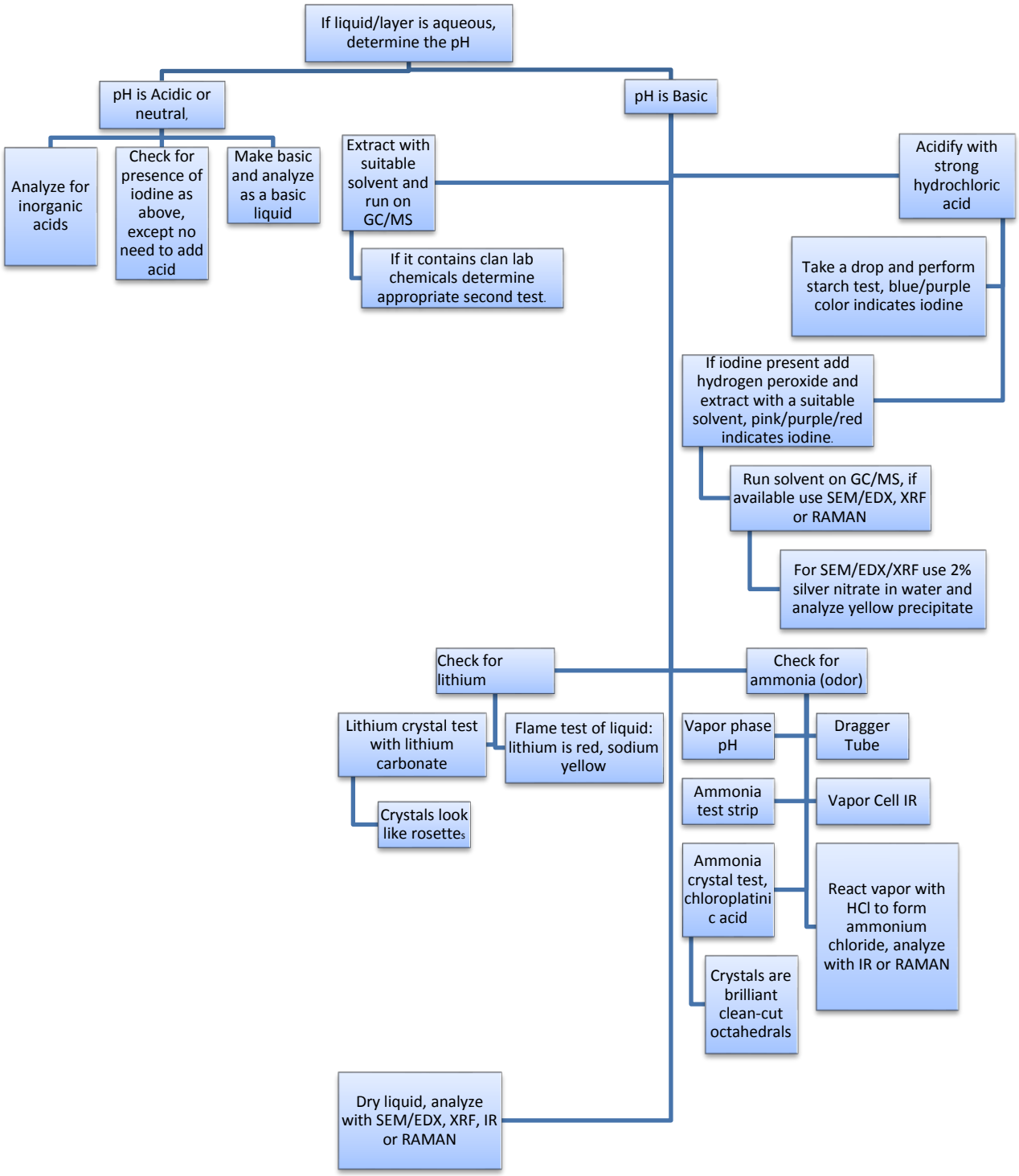
TABLE 2 – Strength of Wording for Results Based on Analytical Technique

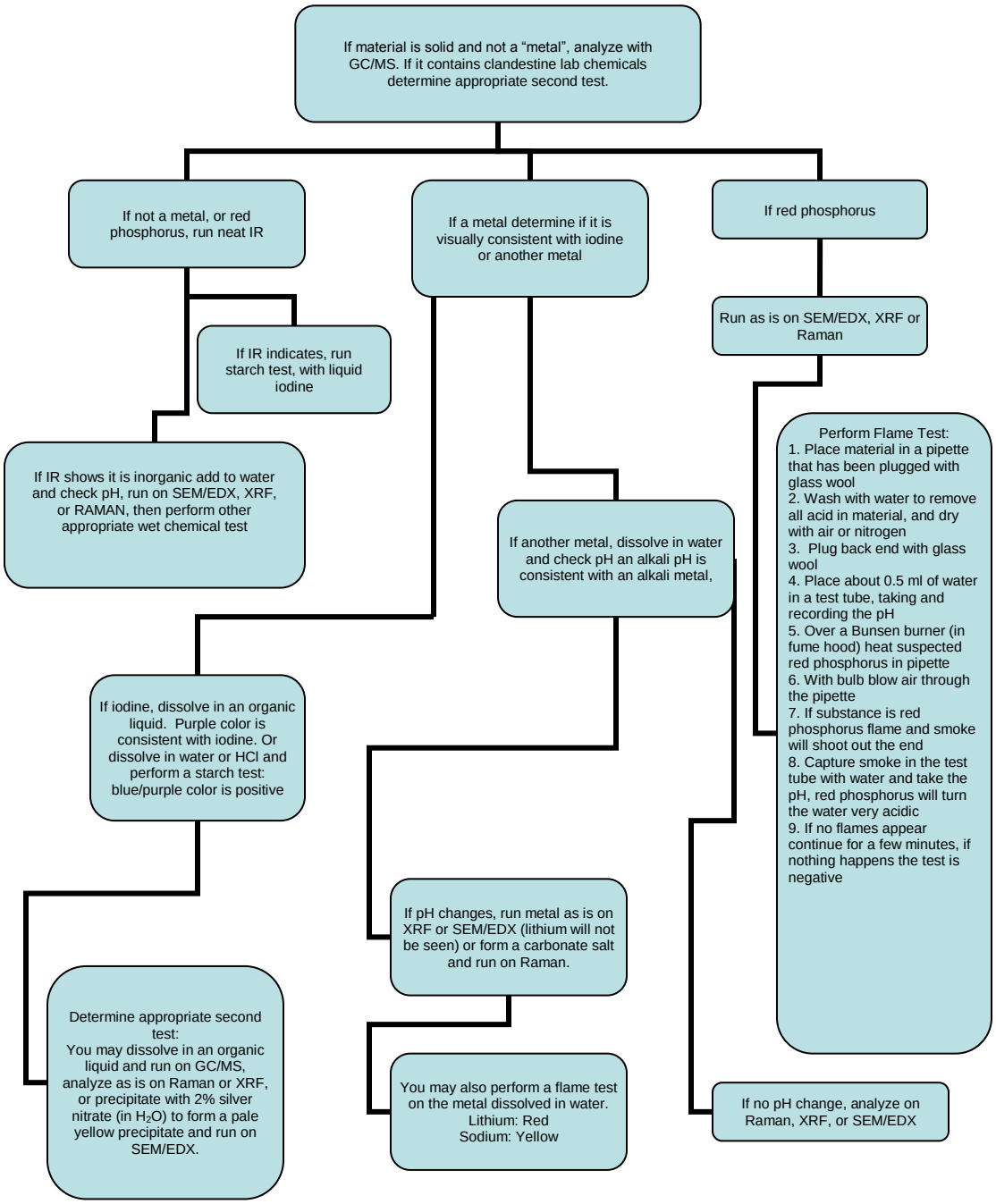
In general, two uncorrelated results are used to identify substances. Consistent with the Seized Drugs section of the technical procedures, each of the available analytical techniques has a certain strength of result that should be communicated in the report. As part of clan lab casework, the analyst relies considerably more on qualifying language compared to controlled substance casework. The student is likewise encouraged to work with the trainer to understand how to construct a final strength of conclusion based on the collection of individual testing results – the final conclusion can be stronger than the “weakest link” provided by the individual testing results. This table summarizes the appropriate strength of result if the technique is included as one of the two tests.

27. APPENDIX H: ANALYTICAL SCHEME

The following flow chart that demonstrates the decision-making in an analytical scheme useful for clan lab evidence. It is important to note that separations, classification tests, and confirmatory analysis are all part of the scheme. As part of clan lab training, the trainee is encouraged to adopt it, adapt it for use consistent with available resources, or to develop another means of demonstrating the logic behind the analysis of evidence.




If liquid/layer is aqueous, determine the pH
pH is Acidic or neutral.
pH is Basic
Analyze for inorganic acids
Check for presence of iodine as above, except no need to add acid
Make basic and analyze as a basic liquid
Extract with suitable solvent and run on GC/MS
If it contains clan lab chemicals determine appropriate second test.
Acidify with strong hydrochloric acid
Take a drop and perform starch test, blue/purple color indicates iodine
If iodine present add hydrogen peroxide and extract with a suitable solvent, pink/purple/red indicates iodine.
Run solvent on GC/MS, if available use SEM/EDX, XRF or RAMAN
For SEM/EDX/XRF use 2% silver nitrate in water and analyze yellow precipitate
Check for lithium
Check for ammonia (odor)
Lithium crystal test with lithium carbonate
Flame test of liquid: lithium is red, sodium yellow
Crystals look like rosettes
Vapor phase pH
Dragger Tube
Ammonia test strip
Vapor Cell IR
Ammonia crystal test, chloroplatinic acid
React vapor with HCl to form ammonium chloride, analyze with IR or RAMAN
Crystals are brilliant clean-cut octahedrals
Dry liquid, analyze with SEM/EDX, XRF, IR or RAMAN


If material is solid and not a "metal", analyze with GC/MS. If it contains clandestine lab chemicals determine appropriate second test.
If not a metal, or red phosphorus, run neat IR
If a metal determine if it is visually consistent with iodine or another metal
If red phosphorus
If IR indicates, run starch test, with liquid iodine
Run as is on SEM/EDX, XRF or Raman
If IR shows it is inorganic add to water and check pH, run on SEM/EDX, XRF, or RAMAN, then perform other appropriate wet chemical test
If another metal, dissolve in water and check pH an alkali pH is consistent with an alkali metal,
Perform Flame Test:
1. Place material in a pipette that has been plugged with glass wool
2. Wash with water to remove all acid in material, and dry with air or nitrogen
3. Plug back end with glass wool
4. Place about 0.5 ml of water in a test tube, taking and recording the pH
5. Over a Bunsen burner (in fume hood) heat suspected red phosphorus in pipette
6. With bulb blow air through the pipette
7. If substance is red phosphorus flame and smoke will shoot out the end
8. Capture smoke in the test tube with water and take the pH, red phosphorus will turn the water very acidic
9. If no flames appear continue for a few minutes, if nothing happens the test is negative
If iodine, dissolve in an organic liquid. Purple color is consistent with iodine. Or dissolve in water or HCl and perform a starch test: blue/purple color is positive
If pH changes, run metal as is on XRF or SEM/EDX (lithium will not be seen) or form a carbonate salt and run on Raman.
Determine appropriate second test:
You may dissolve in an organic liquid and run on GC/MS, analyze as is on Raman or XRF, or precipitate with 2% silver nitrate (in H_2O) to form a pale yellow precipitate and run on SEM/EDX.
You may also perform a flame test on the metal dissolved in water.
Lithium: Red
Sodium: Yellow
If no pH change, analyze on Raman, XRF, or SEM/EDX

CLANDESTINE LABORATORY TRAINING CHECKLIST - PAGE 1

Trainee:		Trainer:		
		Trainee Initials/Date	Trainer Initials/Date	Time for Completion
History of methamphetamine				
	Reading			
	Study/discussion exercises			
Introduction to clandestine laboratory analysis				
	Reading			
	Study/discussion exercises			
Ephedrine and pseudoephedrine				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Methamphetamine via the Hydriodic Acid Reaction				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Methamphetamine via the Dissolveing Metal/Ammonia Reduction				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Other methods of methamphetamine synthesis				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Methcathinone				
	Reading			
	Study/discussion exercises			
	Practical exercises			
3,4-Methylenedioxyamphetamine (MDA) & 3,4-Methylenedioxymethamphetamine (MDMA)				
	Reading			
	Study/discussion exercises			
	Practical exercises			

CLANDESTINE LABORATORY TRAINING CHECKLIST - PAGE 2

Trainee:		Trainer:		
		Trainee Initials/Date	Trainer Initials/Date	Time for Completion
Phencyclidine (PCP)				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Lysergic Acid Diethylamide (LSD)				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Tryptamines				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Phenethylamine (PEA) Analogs				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Gamma-hydroxybutyrate (GHB)				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Fentanyl and its Derivatives				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Yield Estimates				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Report Writing				
	Reading			
	Study/discussion exercises			
	Practical exercises			

CLANDESTINE LABORATORY TRAINING CHECKLIST - PAGE 3

Trainee:		Trainer:		
		Trainee Initials/Date	Trainer Initials/Date	Time for Completion
Testimony				
	Reading			
	Study/discussion exercises			
	Practical exercises			
Competency Examination				
	Study/discussion exercises			
	Practical exercises			