Wilmington Police Department
Crime Laboratory

Forensic Drug Analysis

Standard Operating Procedure

UNCONTROLLED COPY

Table of Contents

1.0 Introduction 4
1.1 Goals 4
1.2 Objectives 4
1.3 Authorization 4
2.0 Operations 5
2.1 Duties, Responsibilities, Accountabilities and Qualifications 5
2.2 Security 5
2.3 Safety 5
2.4 Evidence Control 5
2.5 Case Approach 5
3.0 Training 7
3.1 Introduction 7
3.2 Orientation 8
3.3 Drug Overview 9
3.4 Sample Management 16
3.5 Marijuana Identification Training 18
3.6 Pharmaceutical Preparations Training 21
3.7 Color Test Training 23
3.8 Extractions Training 26
3.9 Fourier Transform Infrared Spectroscopy (FT-IR) Training 29
3.10 Gas Chromatography Mass Spectrometry (GC/MS) Training 33
3.11 Reporting 41
3.12 Courtroom Testimony (Analysis) 42
3.13 Re-training 43
4.0 Instrumentation, Equipment and Supplies 45
4.1 Instrumentation and Equipment 45
4.2 Supplies 45
4.3 Performance Checks and Calibrations 45
4.4 Maintenance 45
5.0 Reagents, Standards and Controls 46
5.1 Reagents 46
5.2 Reference Standards and Materials 46
5.3 Controls 48
6.0 Sample Management 49
6.1 Sample Management Procedure 49
6.2 Safety 49
6.3 Departures from Methods and Procedures 49
6.4 Discovery Procedures 49
7.0 Method 51
7.1 General Analytical Scheme 51
8.0 Procedure 61
8.1 Evidence Control 61
8.2 Evidence Examination 61

UNCONTROLLED COPY

8.3 Sampling 62
8.4 General Analytical Procedure 62
9.0 Parameters 64
9.1 Trace Ultra Gas Chromatograph Operational Parameters 64
9.2 DSQ II Mass Spectrometer Operational Parameters 64
9.3 Spectrum 100 Fourier Transform Infrared Spectrometer Operational Parameters 64
10.0 Quality Control 65
10.1 Introduction 65
10.2 Reagents 65
10.3 Standards 65
10.4 Refrigerators and Freezers 66
10.5 Balances 66
10.6 Gas Chromatograph/Mass Spectrometer (GC/MS) 68
10.7 Fourier Transform Infrared Spectrophotometer (FTIR) 69
10.8 Proficiency Testing 70
10.9 Non-conforming Work 71
11.0 Calculations 78
11.1 Balance Accuracy and Precision Check 78
12.0 Reporting 79
12.1 Reviewing the Results 79
12.2 Preparing the Report 79
12.3 Technical Review of the Results 80
12.4 Reporting the Results 80
12.5 Administrative and Technical Review of the Report 81
12.6 Finalizing the Report 81
12.7 Release and Distribution of the Report 81
13.0 Maintenance 82
14.0 References 83
14.1 Statutes 83
14.2 Laboratory Manuals 83
15.0 Appendices 84
15.1 Color Test Reference Tables 84
15.2 Infrared Spectra Reference Table 87
15.3 Mass Spectra Reference Table 91
16.0 Revision Table 93

UNCONTROLLED COPY

1.0 Introduction

Forensic drug analysis (FDA) involves analyzing seized evidence suspected to be or to contain a controlled substance. This standard operating procedure is not all-inclusive, and references other sources where appropriate. It is always the analyst's responsibility to choose the best analytical scheme for each individual case. It is expected that supervisors and/or colleagues are consulted for extraordinary procedures.

1.1 Goals

To provide timely and high quality analysis of controlled substances

To provide scientifically sound expert testimony on the accuracy and reliability of forensic drug chemistry testing

To function as a resource on forensic drug chemistry to the department and the community

1.2 Objectives

To complete forensic drug analysis within 30 days from the receipt of the request for analysis

To provide rush analysis on a case-by-case basis upon valid request

1.3 Authorization

The methods and procedures in this manual follow guidelines recommended by the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) to perform forensic drug analysis. These are accepted methods of testing in the forensic scientific community. Each individual analyst performing the tests is qualified to perform the duties for forensic drug analysis and is required to maintain proficiency in their testing area(s).

UNCONTROLLED COPY

2.0 Operations

2.1 Duties, Responsibilities, Accountabilities and Qualifications

Laboratory personnel performing these procedures are qualified and authorized according to the Quality Management System and are responsible to adhere to all policies and procedures therein.

2.2 Security

Laboratory personnel adhere to security measures detailed in the Quality Manual, Section 2.3.

2.3 Safety

Laboratory personnel maintain the highest level of safety while working in the laboratory. Refer to MSDS catalogs and the Chemical Hygiene Plan prior to use of any chemicals and/or other laboratory equipment.

A list of emergency contact phone numbers is posted in the laboratory.

2.4 Evidence Control

Refer to Quality Manual, Section 2.8 and the Evidence Control SOP, QP102.8 for evidence control procedures.

2.5 Case Approach

The Crime Laboratory allows for the restriction of analysis to key items within a case to maximize the resources of the laboratory. The most significant items in terms of quantity and schedule are prioritized. Consideration is given to the information contained on the Request for Laboratory Examination, to include but not limited to specific charges or types of offense, items unique to a single suspect, the statement of fact and examinations requested and the descriptions of evidence submitted as well as the analyst's visual inspection of the items.

If it becomes apparent that items not analyzed require analysis for successful prosecution, then upon re-submission, that item is prioritized.

2.5.1 "No suspect, information only" requests are discouraged.

2.5.2 Syringes are only analyzed if they are the only item in the case.

2.5.3 Quantitative analyses are not performed.

2.5.4 Avoid handling evidence repeatedly. The material is sampled and immediately sealed.

UNCONTROLLED COPY

2.5.5 In general, residues in drug paraphernalia, cigarettes or cigarette butts are not analyzed when measurable quantities of the associated drugs are included among the items submitted.

2.5.6 Minimize detailed labeling on small items such as very small metal foil packets, plastic bags or plastic bag corners. Label only those tested. If needed, place these in an additional plastic bag which can be sealed, fully labeled and properly documented in the case notes.

2.5.7 Weights may be obtained for limited numbers of specimens within an item depending on the type of criminal charge. Refer to the Weighing SOP, QP102.10.2.

2.5.8 Sampling is employed in all appropriate cases. Refer to the Sampling SOP, QP102.10.1.

2.5.9 When multiple residue items are submitted (without a measurable quantity/item), similar residues may be combined to result in only one GC/MS sample.

UNCONTROLLED COPY

3.0 Training

3.1 Introduction

This Training Module is designed and intended to prepare a Forensic Chemist to be fully qualified to perform Forensic Drug Analysis for the Wilmington Police Department Crime Laboratory. The training module gives the trainee instructions and activities to complete, including references to read and questions to answer. The Forensic Lab Manager or an authorized Forensic Chemist will act as a mentor to oversee the trainee's activities in the assigned module(s). The training mentor reviews all written answers for correctness and discusses any incorrect answers with the trainee. The training mentor determines if incorrect answers require additional reading or training in the topic. The training mentor reviews all practical exercises performed by the trainee to determine if competency has been met. The training mentor initials and dates the trainee's activity checklist in each module as the module's activities are completed.

The Forensic Drug training program is modular. The modules may be completed in any order, at the discretion and guidance of the training mentor. Each module includes the amount of time estimated for completion. After completion of each module, the trainee and the training mentor date and initial the training summary. The mentor submits the completed training module(s) summary and an authorization form to the Quality Manager for review. If all requirements are satisfied, the Quality Manager forwards the form to the Forensic Lab Manager and the Chief of Police for final authorization. The trainee may perform the duties covered in each module once the module is completed and the trainee is authorized. A certification of training is completed by the training mentor at the completion of Modules 3.1 – 3.11.

This training manual assumes the trainee already possesses a thorough knowledge of fundamental scientific techniques, chemical principles, and instrumentation. The training program familiarizes the trainee with the more common controlled substances, cutting agents, precursors, and byproducts currently abused. Street abuse and manufacturing of drugs is constantly changing; therefore, this training program is updated to match current trends. The trainee is encouraged to seek knowledge of popular controlled substances even after this training program is complete.

UNCONTROLLED COPY

3.2 Orientation

The Wilmington Police Department Crime Laboratory consists of two primary analytical sections: the Forensic Alcohol section and the Forensic Drug Chemistry section. Both sections activities are directed and coordinated by the Forensic Lab Manager who is responsible for the efficient operation of the laboratory.

Forensic Chemists are trained to function in both sections and are called on to work in either section at a given time. Generally, the training is completed in the forensic blood alcohol section first followed by training in the forensic drug section.

The forensic drug section receives evidence suspected to be or to contain a controlled substance. The forensic drug section is responsible for all activities related to this function, including receiving samples, analysis, reporting, and expert testimony.

All work in the section is performed in compliance with the Wilmington Police Department Crime Laboratory Quality Manual, Quality and Technical Procedures, the Property and Evidence Section Manual, the Wilmington Police Department Policy Manual, the City of Wilmington Policies and Directives and all applicable statutes of the North Carolina General Statutes.

This module should be completed in one week.

3.2.1 Activities

a. Read Forensic Drug Analysis SOP, TP102, 1.0 Introduction and 2.0 Operations.

UNCONTROLLED COPY

3.3 Drug Overview

This module seeks to familiarize the trainee with the different classes of drugs of abuse, simple pharmacology of major drug classes, and the basic chemistry of the most commonly abused drugs. This module also familiarizes the trainee with legal aspects related to controlled substances to include scheduling in the North Carolina Controlled Substances Act and the Federal Drug Control Act.

This module should be completed in three weeks.

3.3.1 Activities

a. Review External Document, VA DFS, Controlled Substances Procedures Manual, Sections 6 – 7 and 17 – 30 for use as reference throughout the training program.

b. Read the following references (as available):

1) *Drugs of Abuse*, DEA Publication (available annually at www.justice.gov/dea)

2) *North Carolina General Statues Ch. 90, Art. 5*, "North Carolina Controlled Substances Act" (with emphasis on §90-87; §90-89 to §90-94)

3) Course material from "Introduction to Drug Chemistry." West Virginia University (Online) or enroll in online course if available.

c. Review the following references (as available):

1) Marnell, Tim, editor. *Drug Identification Bible, 2010 Edition.*

2) Ciolino, Laura A. et al. "The Chemical Interconversion of GHB and GBL: Forensic Issues and Implications", *Journal of Forensic Sciences*, 2001; 46(6): 1315-1323.

3) Goodman Gilman, Alfed, M.D., PhD. *The Pharmacological Basis of Therapeutics*. New York: McGraw Hill. 2006.

4) Gross, Susan T. "Psychotropic Drugs in Developmental Mushrooms: A Case Study Review", *Journal of Forensic Science*, 2002; 47(6): 1-5.

5) Levine, Barry. *Principles of Forensic Toxicology*. American Association for Clinical Chemistry. 1999.

6) *North Carolina General Statues Ch. 90, Art.5A*, "North Carolina Toxic Vapors Act"

UNCONTROLLED COPY

7) *North Carolina General Statues Ch. 90, Art.5B*, "Drug Paraphernalia"

8) *North Carolina General Statues Ch. 90, Art.5D*, "Control of Methamphetamine Precursors"

9) United Nations on Drugs and Crime (UNODC). "Recommended Methods for the Identification and Analysis of Cocaine in Seized Materials", New York (2012), pp. 1-18.

10) U.S. Controlled Substances Act, Title 21, Chapter 13 (available online at www.deadiversion.usdoj.gov)

11) Vose, James et al. "Detection of Gamma-Butyrolactone (GBL) as a Natural Component in Wine", *Journal of Forensic Science*, 2001;46(5):1164–1167.

d. Answer the questions below using any available references:

1) Define the following terms:

- Controlled substance
- Distribution
- Manufacture
- Drug
- Narcotic drug
- Marijuana
- Cocaine base
- Hashish and hashish oil
- Anabolic steroid
- Depressant
- Stimulant
- Alkaloid

2) Match the following drugs with their classification and North Carolina scheduling:

Classifications: AS – Anabolic steroid; D – Depressant; H – Hallucinogen; N – Narcotic/Opiate; S – Stimulant

UNCONTROLLED COPY

Drug	Classification	NC Scheduling
3,4-MDMA		
PCP		
Heroin		
Hydromorphone		
Psilocyn		
Methadone		
Pentobarbital		
Salicylamide		
Codeine		
Nandrolone Decanoate		
Methamphetamine		
Caffeine		
Diazepam		
Cocaine HCl		
Dextropropoxyphene		
Meperidine		
Phentermine		
Chlordiazepoxide		
Marijuana		
Oxycodone		
Methylphenidate		
GBL		
Mephedrone		
LAMPA		
Ethanol		
Benzocaine		
Alprazolam		

3) List the physiological effects of the following:

- Designer drug
- Depressant
- LSD
- Anabolic steroids
- Phenethylamines
- Morphine
- Analgesics

4) List the pharmacological actions of the following drug classes:

- Depressants

UNCONTROLLED COPY

- Hallucinogens
- Narcotics
- Stimulants

5) Depressants

   a. What is the difference between a sedative and a hypnotic?

   b. What is the largest group within the depressants?

   c. How are barbiturates classified?

   d. Draw the general structure of a barbiturate.

   e. How are most depressants illegally obtained?

   f. Why are the benzodiazepines included with the depressants? Give their general structure.

   g. What is chloral hydrate and how is it used?

   h. What does synergism mean?

   i. Explain the relationship between GHB, GBL and 1,4-butanediol.

   j. Describe the equilibrium formed between GHB and GBL in aqueous solutions of various pH values. How does this affect the analysis?

6) Hallucinogens

   a. What medicinal use do hallucinogens have?

   b. From what is LSD derived?

   c. What is the chemical name for LSD?

   d. What is peyote? Is it controlled?

   e. What is the scientific name for "magic" mushrooms?

   f. What is the chemical name for MDA? For MDMA? For MDE?

UNCONTROLLED COPY

g. What is the chemical name for PCP? How are the letters of PCP derived from the chemical name?

h. Describe a synthetic route for the clandestine manufacture of PCP.

i. What is the legal use of PCP?

j. What are the chemical names for DMT and STP?

k. What is the structural similarity between STP and MDA?

l. Describe the appearance of the *Salvia divinorum* plant. How is it scheduled?

7) Narcotics

a. Define a narcotic according to North Carolina General Statues.

b. From what plant is opium obtained? How? Where is the major crop grown?

c. What is the definition of an opiate?

d. What are the two classifications of opium alkaloids and how do they differ?

e. What percentage (by weight) of opium are alkaloids?

f. How many alkaloids are there in opium and which is the principal constituent?

g. Name the principal narcotic drugs.

h. What is the chemical name for heroin? Street names?

i. Define and give examples of each:

- Natural opiate
- Synthetic narcotic
- Semi-synthetic narcotic

j. How are narcotics used or administered?

UNCONTROLLED COPY

8) Stimulants

   a. What are the two most common stimulants?

   b. Draw the structure of phenethylamine.

   c. What are the major uses of amphetamines?

   d. How is the word "amphetamine" derived?

   e. Name some amphetamine-related stimulants.

   f. Describe three different synthesis methods for methamphetamine.

   g. What is an anorectic drug?

   h. What are some street names for some commonly encountered stimulants?

   i. When is cocaine classified as a stimulant? As a narcotic?

   j. From what plant is cocaine obtained from? Where is the major crop grown?

   k. How is cocaine base produced from cocaine hydrochloride? How does "crack" differ from "freebase"?

   l. How are various stimulants used or administered?

9) Miscellaneous

   a. What is physical dependence and how does it vary from psychological dependence?

   b. What is meant by tolerance?

   c. What are some common household items with a high potential for abuse?

   d. Define the following drug actions:

UNCONTROLLED COPY

- analgesic
- antipyretic
- antitussive
- tranquilizer
- antichlorinergic
- vasoconstrictor
- antihelmintic
- diuretic
- bronchodilator
- antibiotic
- vitamin
- anesthetic

e. What is the difference between an antidepressant and a stimulant?

f. Name four common tricyclic antidepressants.

g. What is the difference between an anabolic steroid and a corticosteroid?

10) Define the schedules in the North Carolina General Statues and criteria for placing a drug in each.

11) Describe the following terms as if you were addressing a lay audience or jury panel:

- Stimulant
- Anesthetic
- Antibiotic
- Hallucinogen
- Designer drug

e. Develop a "Drug Known" notebook for each drug listed in Appendix 15.2. It is most helpful to do color tests by drug group so that differences in chemical structure can be correlated to different test results. Fill out the drug name, schedule information, pharmacological information and structure. The list of drugs used to develop the notebook can be modified based on the availability of drug standards in the laboratory and recommendations from the training mentor.

UNCONTROLLED COPY

3.4 Sample Management

This module familiarizes the trainee with general laboratory procedures pertaining to forensic drug testing, the general analytical scheme and sampling plan when testing for controlled substances, and weighing practices associated with forensic drug testing. The trainee reviews the established laboratory procedures for sample management and with guidance from the training mentor addresses each unique sample situation individually.

This module should be completed in three weeks.

3.4.1 Activities

a. Read Forensic Drug Analysis SOP Sections 4.0 – 8.0, 10.1-10.3 and Sampling SOP, QP102.10.1.

b. Review the following references (as available):

1) European Network of Forensic Science Institutes Drugs Working Group. "Guidelines on Representative Sampling" 2002-2003.

2) Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations. 3rd Edition, 2007 – 08 – 09.

c. Observe training mentor receive a forensic drug sample, to include identification of the proper analytical scheme.

d. Read Weighing SOP, QP102.10.2 and Forensic Drug Chemistry SOP Section 10.4.

e. Perform the procedure outlined in Forensic Drug Chemistry SOP Section 10.5.2.

f. Observe the training mentor weigh a forensic drug sample and determine an appropriate sample for analysis.

g. Answer the questions below:

1) What is a primary drug standard? What is a secondary drug standard? Who must observe the receipt of a primary drug standard or transfer of a secondary drug standard?

2) How is a laboratory report number assigned for forensic drug samples?

3) What should be done if a discrepancy is found with the request form during inventory of the forensic drug sample?

UNCONTROLLED COPY

4) What are the three general analytical schemes outlined in the Forensic Drug Analysis SOP?

5) The laboratory has received an item of evidence with seven visually homogeneous sub-items of a suspected controlled substance. Under the administrative sampling plan how many of the sub-items should be analyzed for charges of simple possession? For charges of possession with intent to distribute? If the District Attorney has requested additional analysis how many of the sub-items should be analyzed under the hypergeometric sampling plan?

6) How can residues be sampled? How should residue samples be returned to the evidence?

7) List the analytical techniques used in the laboratory and the corresponding category A, B, or C.

8) What are the minimum standards that must be met in order for an identification of a drug or chemical to be reported?

9) How could the category of an analytical technique be lowered? Give an example that could occur in the laboratory.

10) The analytical balance shows the weight of a sample in the packaging to be 15.279 g, how should that weight be reported? How should it be reported if the balance shows 6.243 g when the sample is weighed without the packaging?

11) The laboratory has received an item of evidence with seven visually homogeneous sub-items of a suspected controlled substance. Under the administrative sampling plan how will the sub-items be weighed for charges of simple possession? For charges of possession with intent to distribute? If the District Attorney has requested additional analysis how will the sub-items be weighed under the hypergeometric sampling plan?

12) The laboratory has received an item of evidence with two hundred and fifty (250) visually homogeneous sub-items of a suspected controlled substance with the charge of trafficking.
    a. What sampling plan should be used? How will the item be sampled and weighed?
    b. How will the weights be reported once analysis is complete? How is the uncertainty of measurement determined for combined weights?

UNCONTROLLED COPY

3.5 Marijuana Identification Training

The standard practice in forensic laboratories for the identification of marijuana is a macroscopic characterization based on leaf shape, a microscopic characterization of the leaf's botanical features, followed by a color test (Modified Duqenois-Levine). The newly trained analyst also has the option of confirmation with a GC-MS analysis for tetrahydrocannabinol (THC). Once properly trained an analyst can positively identify marijuana based on specific botanical features and a positive Modified Duquenois-Levine color test.

This module should be completed in three weeks.

3.5.1 Activities

a. Read Forensic Drug Analysis SOP Sections 7.1-7.1.3 and External Document, VA DFS, Controlled Substances Procedures Manual, Section 6, Marijuana and Hashish Oil.

b. Read Color Test Working Instructions Section 2.1.

c. Read Drug Identification Bible pp 386-407 and review pp 581-686.

d. Read the following references (as available):

1) *Marijuana*, DEA Training Manual.

2) Fisher, Deborah L. and Thornton, John I. "The Duquenois Reaction and Phenols of the Cannabinoid and Non-cannabinoid Type" *CAC Newsletter,* (1989), April, 17.

3) Nakamura, George R. and Thornton, John I. "The Forensic Identification of Marijuana: Some Questions and Answers" *Journal of Political Science and Administration*, (1973), 1(1), 102-112.

4) Resolution I and II, *American Academy of Forensic Sciences* Criminalistics Section Meeting, (1974), February.

e. Review the following references (as available):

1) Joyce, C.R.B. and Curry, S.H. "The Botany & Chemistry of Cannabis" (1970), J. & A. Churchill, London, 10-38.

2) Small, Ernest. "The Forensic Taxonomic Debate on Cannabis: Semantic Hokum" *Journal of Forensic Science*, (1976), 21 (2), 239-251.

UNCONTROLLED COPY

3) Thornton, John I. and Nakamura, George R. "The Identification of Marijuana" *Journal of the Forensic Science Society*, (1972), 12, 461-503.

4) United Nations on Drugs and Crime (UNODC). "Recommended Methods for the Identification and Analysis of Cannabis and Cannabis Products", New York (2009).

f. Observe training mentor perform analysis of known marijuana sample.

g. Prepare Modified Duquenois-Levine color test reagent.

h. Conduct analysis of known marijuana sample under the supervision of the training mentor.

i. Conduct examination of plant training samples; keep notes in "Drug Known" book.

j. Conduct the Modified Duquenois-Levine Ratio Test.

k. Answer the following questions:

1) What is the definition of marijuana per the North Carolina General Statute?

2) Compare and contrast the definition of marijuana from the North Carolina General Statutes to that in the Federal Controlled Substances Act. What are the possible analytical implications?

3) What is sensimilla (sinsemilla) and how is it grown?

4) Define "agronomic" variety and differentiate between Cannabis sativa, Cannabis ruderalis, Cannabis indica, and Cannabis Americana.

5) What is a cystolithic hair? Do any other plants have cystolithic hairs? If so, name two other plants with cystolithic hairs.

6) Describe and draw a typical marijuana leaf.

7) On which part(s) of the marijuana plant will its characteristic hairs not be found?

8) What parts of the plant contain THC?

9) What should you be looking for when examining an alleged marijuana sample at approximately 10X? At 45X?

UNCONTROLLED COPY

10) If you receive an item submitted as evidence which is suspected black tar heroin, but tests negative for opiates, what other test(s) should be performed?

11) Describe the differences between hashish and hash oil including preparations, schedule, and analysis.

12) You have received a thick brown liquid as evidence. What tests would you perform on it?

13) What is the difference between the "original" and the "modified" Duquenois test? Which one do we use?

14) What is the purpose of the vanillin in the Duquenois reagent?

15) Is hemp the same as marijuana?

16) Do cannabinol or cannabidiol give a positive Duquenois-Levine?

17) What is the major psychoactive compound in marijuana and marijuana preparations?

18) What are the two numbering systems for cannabinoids in use today? Draw THC and show how these numbering systems differ.

19) What types of isomers are Δ9-THC and Δ8-THC? Which is the more stable?

20) Is d- or l-THC the naturally occurring isomer?

21) Are the cannabinoids acidic or basic? Polar or non-polar?

22) Chemically, can any of the other cannabinoids break down or be converted to THC? Does THC break down?

l. Complete the Marijuana/Plant Material Training Exam. Trainee must correctly identify all samples containing marijuana. If the trainee does not correctly identify all samples containing marijuana the training mentor conducts re-training on sections e-i of 3.5.1.

UNCONTROLLED COPY

3.6 Pharmaceutical Preparations Training

This training familiarizes the trainee with the analytical procedures for pharmaceutical preparations and pills. Pharmaceutical preparations possess unique identifying information both in the general appearance of the preparation and the inscriptions or markings. The trainee reviews resources used to identify these unique markings and the methods for further conformational analysis. Any tablets, unmarked pills, or capsules that do not appear to have been manufactured by a legitimate pharmaceutical company, or appear to have been tampered with are analyzed for controlled substances and treated as unknowns.

This training should be completed in three weeks.

3.6.1 Activities

a. Read Forensic Drug Analysis SOP Section 7.1.2 and 7.1.4 and External Document, VA DFS, Controlled Substances Procedures Manual, Section 7.

b. Review the following references (as available):

1) Identadrug, website subscription: http://www.identadrug.com

2) Marnell, Tim, editor. *Drug Identification Bible, 2010 Edition.*

3) Poison Control Center

4) DEA Drug Logo Index, v 3.31.

5) O'Neil, Maryadele J., editor. *The Merck Index, Fourteenth Edition.* Whitehouse Station, N.J.: Merck & Co., Inc., 2006.

6) Rx-ID, electronic version. Amera-Chem Inc., Version 2008.1.

c. Add description and drawing of pharmaceutical preparations to "Drug Known" book.

d. Answer the following questions:

1) Name the manufacturers and active ingredients of the following preparations:

- Desyrel
- Oxycontin
- Adderall
- Demerol
- Preludin

UNCONTROLLED COPY

- Ritalin
- Viagra
- Keflex
- Percodan
- Darvon
- Soma
- Paxil
- Dalmane
- Librax
- Valium
- Fiorinal
- Wellbutrin
- Zoloft
- Vicodin

2) List four possible references for tablet logo identification.

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

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

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

e. Observe training mentor conduct visual identification and analysis of unknown pills or tablets.

f. Conduct visual identification and prepare sample for analysis under the supervision of the training mentor. The trainee must successfully conduct visual identification of pharmaceutical preparations. If the trainee is unsuccessful the mentor will conduct re-training of section e of 3.6.1.

UNCONTROLLED COPY

3.7 Color Test Training

This module familiarizes the trainee with the preparation, storage, and proper handling procedures of color test reagents; the theory and use of chemical color tests, including the advantages, disadvantages, and limitations. Color testing is a presumptive analysis used to assist the analyst with further confirmative testing. Although a limited number of color tests are used in the laboratory regularly, this training covers a large number of color tests to familiarize the trainee with available options for future use in casework.

This module should be completed in three weeks.

3.7.1 Activities

a. Read Forensic Drug Analysis SOP Sections 7.1.5 and 10.2

b. Read Color Tests Working Instructions.

c. Read Reagent Check SOP, QP102.1.

d. Read the following references (as available):

1) National Institute of Justice. "Color Test Reagents/Kits for Preliminary Identification of Drugs of Abuse", *NIJ Standard – 0604.01*, U.S. Department of Justice, July 2000.

2) Moffat, Anthony C et al., editors. *Clarke's Analysis of Drugs and Poisons.* London: The Pharmaceutical Press, 2004, pp. 279-300.

3) O'Neal, Carol L. et al. "Validation of Twelve Chemical Spot Tests for the Detection of Drugs of Abuse", *Forensic Science International*, 2000, pp. 189-201.

4) Course material from "Chemical Spot Tests for Illicit Drugs." West Virginia University (Online) or enroll in online course if available.

e. Prepare color test reagents for the following: (1) Marquis, (2) Cobalt Thiocyanate, (3) Sodium Nitroprusside.

f. Perform the following color tests on all available drug standards (using secondary standards when possible) or as directed by the training mentor: (1) Marquis, (2) Cobalt Thiocyanate, (3) Sodium Nitroprusside. Annotate results in "Drug Known" book; add additional color tests information not currently used for reference.

g. Answer the following questions:

UNCONTROLLED COPY

1) For each color test used in this section, list the formulation, types of compounds that react with each test, and state what reaction would be observed.

2) Describe as to a jury how a color test is performed, including the purpose and value of the test.

3) An officer calls stating that the field test kit used on a submitted sample indicated the presence of heroin. Your analysis reveals no controlled substances. How might you explain this?

4) When and how often is the reliability of the color test reagents checked?

5) What is the approximate sensitivity of each color test you are using?

6) Briefly describe the mechanisms of the following color tests:

   - Marquis
   - Cobalt thiocyanate

7) Describe the difference between the terms "sensitivity" and "selectivity" as they relate to color tests.

8) What color test reagents are light sensitive, or are subject to thermal or temporal deterioration?

9) Define "false positive". Give three examples of false positive color tests.

10) Define "false negative". Give three examples of false negative color tests.

11) Describe the use of blanks pertaining to spot tests.

12) What effect do mixtures have on spot test results?

13) What effect does time have on color test reagents?

14) Define the following terms:

   - Precipitate
   - Complex
   - Ligand
   - Coordination Number

15) What are common impurities found in Cocaine HCl and cocaine Base?

UNCONTROLLED COPY

16) Why do cocaine hydrochloride and cocaine free base react differently with the cobalt thiocyanate test?

17) Which of the color tests will be useful for screening for opiates?

18) What is the basic structure of an opiate?

19) What are common impurities found in Heroin?

20) Which of the color tests will be useful for screening for scheduled phenethylamines?

21) What is the basic structure of a phenethylamine?

22) Is an orange-to-brown Marquis test specific for Methamphetamine? For phenethylamine derivatives?

23) Is methamphetamine a primary, secondary, or tertiary amine? What about amphetamine?

24) What are common impurities found in methamphetamine?

25) What are some of the drugs, other than LSD, that have been found on blotter paper?

26) Are hormones the same as steroids? Explain.

27) What is a milligram? (Define in simple terms, as for a jury.)

28) Rarely will a pure drug sample be encountered in casework. What is an excipient? An adulterant? A diluent? Name some examples.

UNCONTROLLED COPY

3.8 Extractions Training

This module familiarizes the trainee with sample extraction methodology. Currently the crime laboratory does not conduct quantitative analysis of forensic drug samples which limits the need for extensive extractions. For qualitative, the gas chromatograph separates the analyte of interest from additional components in a sample. However, some extraction procedures may be necessary at times. This module serves to provide the trainee with resources and an overview of extraction procedures.

This module should be completed in three weeks.

3.8.1 Activities

a. Read Extractions Working Instructions.

b. Review The Merck Index for solvents of commonly submitted drugs.

c. Add information useful for extractions to "Drug Known" book (ie. Solvents soluble in, acid/basic/neutral, typical extraction schemes for sample type, etc.)

d. Review commonly used extraction methods with training mentor.

e. Answer the following questions:

1) What is a matrix?

2) Define the following:

- Unsaturated solutions
- Saturated solutions
- Supersaturated solutions
- Azeotrope
- Reflux

3) Define the terms "extraction", "partition", and "salting out". How does changing the volumes of solvents used affect each of them?

4) What is the difference between evaporation and sublimation?

5) What problems may be encountered if ether evaporates to dryness?

6) What is a dry extract?

7) What effect does temperature have on a drug extraction?

UNCONTROLLED COPY

8) Describe how a series of several extractions will be more effective than a single extraction (using the same volume of solvent) considering the concept of partition coefficients.

9) What problems are encountered when using a large volume of solvent during extractions?

10) How can water be removed from organic solvents?

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

12) What does pH stand for? pK_a?

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

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

15) What does amphoteric mean?

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

17) What is ion pairing? Diagram how it works using equilibrium considerations.

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

19) What separation advantages does chromatography have over extraction procedures? Disadvantages?

20) Describe how a soxhlet extractor works.

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

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

23) How is morphine best extracted from powder form?

24) How would you analyze blotter paper suspected to contain LSD? Why is the blotter paper soaked in the dark prior to extraction?

UNCONTROLLED COPY

25) Given a tobacco cigarette that you suspect has been dipped in a PCP solution, how would you clean it up for quantitation and analysis? What is the purpose of the H_2O wash?

26) How would you "clean up" a neutral drug?

UNCONTROLLED COPY

3.9 Fourier Transform Infrared Spectroscopy (FT-IR) Training

This module familiarizes the trainee with the theory of the Infrared Spectrometer (IR) and its practical applications in forensic drug analysis. The trainee uses theoretical knowledge gained through research and hands-on experience with the training mentor to prepare to utilize this instrument for forensic drug analysis in casework.

This module should be completed in four weeks.

3.9.1 Activities

a. Read Forensic Drug Analysis SOP Sections 7.1.7, 9.3, 10.7 and Maintenance Plan SOP, QP102.5.

b. Read the following references (as available):

1) Drake, A. (2004). Infra-red Spectroscopy. In A.C. Moffat (Ed.), *Clarke's Analysis of Drugs and Poisons,* (p. 328-345), London: The Pharmaceutical Press.

2) PerkinElmer. (2005). Technical Note: FT-IR Spectroscopy Attenuated Total Reflectance (ATR). Shelton, Connecticut. Retrieved July 10, 2007 from http://las.perkinelmer.com/content/technicalinfo/tch_ftiratr.pdf

3) PerkinElmer. (2004). Technical Note: ATR Accessories An Overview. Shelton, Connecticut. Retrieved July 10, 2007 from http://las.perkinelmer.com/content/technicalinfo/tch_atraccessories.pdf

4) "Spectrum 100 User Guide" and "Spectrum Software User Guide." Perkin Elmer. (2010). Spectrum Manuals CD (L1050002-O).

5) "Universal ATR" Tutorial. Perkin Elmer. (2010). Spectrum Multimedia CD (L1050005-B).

c. Answer the following questions:

1) Describe in simple terms, as for a jury, how the FT-IR works.

2) Describe in scientific terms how the FT-IR works and the theory behind FT-IR spectroscopy.

3) Describe the electromagnetic spectrum.

a. What is the upper and lower limit on the infrared region of the electromagnetic spectrum?

UNCONTROLLED COPY

b. What region is the most useful analytically?

c. What is the standard range of most instruments?

4) Define the following terms:

- Wave
- Wavelength
- Wavenumber
- Frequency
- Dipole moment
- Absorption
- Transmittance
- Overtone
- Harmonic vibration
- Combination band
- Fundamental vibration
- Monochromator
- Interferometer
- Homonuclear
- Amplitude
- Centerburst

5) What is "Fourier Transform" and how does it apply to IR?

6) Explain the theory behind the Attenuated Total Reflectance (ATR) sampling unit including the differences between single-bounce and multi-bounce units.

a. Describe any differences in the spectra obtained using ATR vs. regular transmittance.

b. Explain the function of the ATR correction within the software including when it is permissible to use a corrected spectra in case work.

7) What is meant by the "fingerprint region" of an IR spectrum? Why is it significant?

8) Can IR differentiate optical isomers? Diastereomers? Structural isomers?

9) Which organic functional groups correspond to the following absorption frequencies?

UNCONTROLLED COPY

- 3639-3633 cm^{-1}
- 2990-2850 cm^{-1}
- 1650-1510 cm^{-1}
- 1750-1740 cm^{-1}
- 770-690 cm^{-1}
- 760-540 cm^{-1}

10) What two conditions must be met in order for infrared absorption to occur?

11) Explain Beer's Law.

12) What are the two basic categories of molecular vibration?

13) What are the four types of bending?

14) What is meant by vibrational coupling?

15) Which will vibrate with higher frequency, C-H bond or a C-C bond and why?

16) What does hydrogen bonding do to the vibrational frequency of a hydroxyl or an amine group?

17) Describe the absorptions for the following groups:

- -O-H
- -N-H
- >C=O
- -C-O-
- -C-H
- -C≡N
- $-NO_2$
- Aromatic Substitutions

18) What is polymorphism and how does it influence IR spectra?

19) What model IR does our laboratory use?

    a. What radiation sources and detectors are used in the FTIR and its attachments in our laboratory?

20) What causes a sloped baseline?

UNCONTROLLED COPY

21) Explain baseline correction and how it is performed.

22) What is spectral subtraction and under what conditions is it possible?

23) What are the differences between background subtraction and spectral subtraction?

24) What is the relationship between resolution and data point spacing?

25) What resolution are samples normally run in our laboratory?

26) What computer libraries are available in our laboratory and what are the resolutions of the spectra contained in them? What other libraries are available?

27) Describe how a spectrum is auto-saved and/or saved.

28) Describe how ATR analysis can be run on powders, liquids, and mixtures.

29) What are the advantages/disadvantages of a GC/MS compared to an IR when used for identification purposes?

30) Describe the preventative maintenance schedule and the QA/QC procedures performed on the IR including the software.

31) At what wavenumbers does the diamond crystal absorb in an ATR spectrum?

d. Observe training mentor prepare sample, conduct analysis, and interpret analysis on FTIR.

e. Analyze the following drug standards (using secondary where available) using FTIR:

- Methamphetamine
- Amphetamine
- Cocaine HCl
- Cocaine Base
- Others identified by training mentor

Match spectra to WPCL data base or other literature sources. Annotate key peaks in "Drug Known" book.

UNCONTROLLED COPY

3.10 Gas Chromatography Mass Spectrometry (GC/MS) Training

This module familiarizes the trainee with the theory of Gas Chromatography Mass Spectrometry (GC/MS) and its practical applications in forensic drug chemistry. The trainee uses theoretical knowledge gained through research and hands-on experience with the training mentor to prepare to utilize this instrument for forensic drug analysis in casework.

This module should be completed in four weeks.

3.10.1 Activities

a. Read Forensic Drug Analysis SOP Sections 7.1.6, 9.1 – 9.2, 10.6 and Maintenance Plan SOP, QP102.5.

b. Read the following references (as available):

1) Watson, D. (2004). Mass Spectrometry. In A.C. Moffat (Ed.), *Clarke's Analysis of Drugs and Poisons,* (p. 379-391), London: The Pharmaceutical Press.

2) Dawling, S. (2004). Gas Chromatography. In A.C. Moffat (Ed.), *Clarke's Analysis of Drugs and Poisons,* (p. 425-499), London: The Pharmaceutical Press.

3) Trace DSQII and XCalibur Software User Manuals. Electronic files located on GC/MS computer.

c. Review the following references (as available):

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

d. Answer the following questions:

1) Describe in simple terms, as for a jury, how a gas chromatogram works as it relates to forensic drug analysis.

2) What affect do the following have on retention time:

- Concentration
- Other compounds in the sample
- Free base/acid form vs. salt form

3) Why does the general drug screen method not collect data before four minutes?

4) Describe the proper manual injection technique.

UNCONTROLLED COPY

5) What factors govern the amount of sample to be injected? How much sample/component can the average capillary column hold? What factors influence this?

6) What temperature should the injection port be under normal circumstances and why?

7) Describe the "solvent effect."

   a. How is it done and why is it important?

   b. What factors affect the efficiency of the solvent effect?

8) What types of compounds should be included in a test mixture used to assess chromatographic performance? Why would these compounds be included and what would each be designed to evaluate?

9) What type(s) of GC(s) (model, manufacturer, etc.) does the drug laboratory use?

   a. What type(s) of injection ports, carrier gases, flows, columns, and detectors does each GC incorporate?

10) Outline a logical troubleshooting schematic for isolating the source of a GC system problem.

11) Describe the preventive maintenance schedule and QA/QC procedures performed on the GC.

12) Explain how derivatization is performed, including why it is used sometimes for analysis.

13) If two drug compounds were to co-elute on the GC, what could be done to resolve the peaks?

14) What is mass spectrometry?

15) Describe the theory behind its use as an identification technique.

16) What types of information are obtained from a GC/MS?

17) Define the following terms:

- Relative abundance

UNCONTROLLED COPY

- Base peak
- Molecular ion
- Quasimolecular ion
- Parent/precursor ion
- Daughter/product ion
- Mass/charge ratio
- Mass spectrum
- Unit mass resolution
- Normalization
- DFTPP Normalization
- Carbonium ion
- Cleavage
- AMU
- Isobaric
- Radical
- Doubly charged ion
- Calibration compound
- Torr
- Atmosphere
- Total Ion Current

18) What is a "metastable peak"? When and where does is occur?

19) What is the sensitivity of a GC/MS?

20) What is the difference between spectrometry and spectroscopy?

21) Why can column bleed cause a problem in GC/MS and how is it corrected? Septum bleed?

22) What is the most common mode of ionization?

23) Find a diagram of the E.I. source for the Trace DSQII.

    a. Are the ions formed positive or negative?

    b. Do they have an even or odd number of electrons?

    c. What is the ionization efficiency of this technique?

    d. What governs the relative abundance of the ions formed?

UNCONTROLLED COPY

24) What governs the number and energy of the electrons emitted by the filaments?

25) From what are the filaments made?

26) What is an "ionization appearance potential" curve?

   a. What is the usual electron energy used in an E.I. source for complete ionization and why?

   b. What effect does variation in this energy have on ion abundance?

   c. If a molecule is ionized with energy just at its appearance potential, what information may be obtained?

27) What vacuum conditions are necessary in the ionization source and the analyzing regions of an MS and why?

   a. Describe how a rough pump works.

   b. Describe how a diffusion pump works.

   c. Describe how a turbomolecular pump works.

   d. Is it necessary that the vacuum remain constant?

28) What temperature conditions must be maintained in the ion source?

29) Describe how the ions are accelerated once they are formed.

30) Explain how chemical ionization is performed.

   a. What are its advantages/disadvantages with respect to electron ionization?

   b. What is the number of fragment ions produced by this method dependent on?

   c. Do the ions formed by this process have an even or odd number of electrons?

31) Describe how a quadrupole mass analyzer works.

   a. What factors influence the practical limits of the quadrupole as a mass filter?

UNCONTROLLED COPY

b. What determines whether an ion will have a stable trajectory through the quadrupoles?

32) Define mass resolution.

a. What does a resolution of 500 mean?

b. What is the resolution a function of?

c. Is the instrument in the laboratory a low, medium or high resolution instrument?

d. What resolution values are associated with these terms?

33) Describe how an electron multiplier works.

34) Why is the electron multiplier the detector of choice?

a. What are the limiting factors as to how well an electron multiplier can detect incoming ions?

35) List what the base peaks and molecular ions are for each of the following:

- Cocaine
- Heroin
- Phencyclidine
- LSD
- Methamphetamine

36) Can ephedrine and pseudophedrine be distinguished by MS?

37) Can optical isomers and diastereomers be differentiated via MS?

38) Obtain a mass spectrum for cocaine and account for the major peaks in the spectrum.

39) Obtain literature mass spectra of the diastereomers of cocaine and discuss the differences.

40) List the isotopic abundances for each of the following elements: H, C, N, O, F, Si, P, S, Cl, Br, I

41) What is the nitrogen rule?

UNCONTROLLED COPY

42) If a molecular formula has been determined, how can the number of rings and double bonds be determined?

43) What is the "index of hydrogen deficiency"?

44) What influences what bond sites will be ruptured to create molecular fragments?

45) Describe how fragmentation patterns are influenced by:

- Branched carbon atoms
- Double bonds
- Rings
- Hetero-atoms
- Carbonyl groups

46) What are the M+2 (or A+2) elements?

47) What percentage of intensity of a molecular ion is contributed to the M+1 peak by carbon atoms?

    a. What is the formula for calculating the number of carbon atoms in a molecule?

    b. How can the M+1 peak be used to determine the molecular weight?

48) What requirements are necessary for an ion to be considered a molecular ion?

    a. Define the term "logical neutral loss" and give examples.

    b. What mass losses during fragmentation are highly unlikely?

49) What is the most desirable characteristic of mass spectra of trimethylsilyl derivatives?

50) In what types of compounds is a molecular ion peak frequently not detectable?

51) In what types of compounds are molecular ion peaks most likely to occur?

52) What do the peaks occurring at higher mass numbers than the molecular ion often represent?

53) Describe the isotope pattern for Cl and Br.

UNCONTROLLED COPY

54) What ions can be associated with the following m/e ratios?

- 43
- 58
- 77
- 91

55) Define the following terms and describe how these terms relate mass spectrometry to chromatography.

- scan rate
- scan cycle time
- reset time
- a/d conversion rate
- spectral tilting
- Mass peak detect threshold
- GC peak detect threshold

56) Describe how to perform the following techniques:

- Headspace analysis
- Wet needle injection

57) Describe the preventive maintenance schedule and the QA/QC procedures performed on the GC/MS.

58) Describe the acceptance criteria needed for using retention time data from GC/MS runs.

59) Describe the use of blanks on the GC/MS.

60) Explain, as to a jury, how a mass spectrometer operates and is applied in forensic drug analysis.

e. Perform an autotune on the GC/MS and describe what each value on the report represents. What types of parameter values may indicate a problem with the instrument?

f. Compare the mass spectral data for ephedrine, pseudoephedrine, methamphetamine, and propoxyphene. What are the significant differences which make these spectra unique to their parent compound?

g. Compare the mass spectra for LSD and LAMPA and indicate their differences.

UNCONTROLLED COPY

h. Obtain an unknown spectrum from your training mentor. Using interpretive methods, give as much information about the unknown compound as possible. Review findings with training mentor. Training mentor will conduct re-training if interpretation is inadequate.

i. Perform a headspace injection of a mixture of volatile solvents.

j. Obtain unknown drug samples (at least one of each: powder, pill/tablet, rock, plant material) from your training mentor. Analyze these samples using any and all methods qualified to perform. Report results to training mentor. The trainee must correctly identify if a controlled substance is present and the identity of that controlled substance. The trainee must present sufficient data to the training mentor to support his or her conclusions. The training mentor determines if the trainee made the appropriate conclusions about each sample. The training mentor conducts re-training as necessary.

UNCONTROLLED COPY

3.11 Reporting

Reporting describes the process of technical review of results, Sample Information Log entry, analyst verification, and administrative review of reports prior to release. This module familiarizes the trainee with these reporting procedures for forensic drug analysis.

This module should be completed in two weeks.

3.11.1 Activities

a. Read Forensic Drug Analysis SOP Section 12, Quality Manual 2.10 and Reporting Results SOP, QP102.10.

b. Observe the training mentor or qualified analyst perform the steps below:

1) Technical review of forensic drug report and supporting documentation
2) Administrative review of forensic drug report
3) Distribution of report

c. Prepare a written report of two unknown samples from 3.10.1q

d. Take the WPCL FDA Uncertainty of Measurement Training

e. Explain the following terms used for reporting:

- Insufficient for Identification
- No controlled substance found
- Inconclusive

UNCONTROLLED COPY

3.12 Courtroom Testimony (Analysis)

A forensic drug chemist provides expert testimony on the theory and operation of forensic drug analysis and the accuracy and reliability of results. The forensic chemist must complete Section 4.3 of the Court Testimony Training SOP, QP102.9.1, and may then testify to the accuracy and reliability of the forensic drug analysis and results.

UNCONTROLLED COPY

3.13 Re-training

In the event that a qualified analyst is hired from an external laboratory, a previously qualified analyst has not performed testing in the Forensic Drug Analysis discipline for more than one year and has not maintained proficiency, or a current analyst fails a proficiency examination; the analyst must participate in a modified training program to ensure competence and proficiency in the area of Forensic Drug Analysis.
Certain training activities are required for an analyst to be authorized or re-authorized to perform the processes in this SOP.

3.13.1 Activities

a. Read Forensic Drug Analysis SOP.

b. Review the references in this training module.

c. Conduct analysis of known marijuana sample under the supervision of the training mentor and correctly identify samples containing marijuana.

d. Conduct visual identification of a pill correctly and prepare sample for analysis under the supervision of the training mentor.

e. Perform the following color tests on all available drug standards (using secondary standards when possible) or as directed by the training mentor: (1) Marquis, (2) Cobalt Thiocyanate, (3) Sodium Nitroprusside.

f. Review commonly used extraction methods with training mentor.

g. Analyze the following drug standards (using secondary where available) using FTIR:

- Methamphetamine
- Amphetamine
- Cocaine HCl
- Cocaine Base
- Others identified by training mentor

Match spectra to WPCL data base or other literature sources. Annotate key peaks.

h. Analyze the drug standards from the previous exercise using GC/MS.

i. Obtain unknown drug samples from your training mentor. Analyze these samples using all necessary methods to be able to meet criteria to report the identity. The trainee must correctly identify if a controlled substance is present and the identity of that controlled substance. The trainee must present sufficient

UNCONTROLLED COPY

data to the training mentor to support his or her conclusions. The training mentor determines if the trainee made the appropriate conclusions about each sample. The training mentor conducts re-training as necessary.

j. Prepare a written report of two unknown samples from the previous exercise.

k. Prepare to be able to answer any questions from this training module in an oral examination.

l. Participate in a moot court exercise. Be able to explain your work in simple (non-scientific) terms.

UNCONTROLLED COPY

4.0 Instrumentation, Equipment and Supplies

4.1 Instrumentation and Equipment

4.1.1 Gas Chromatograph/Mass Spectrometer, Liquid Auto Sampler, Data System and Thermo Gold TR-5MS column 30m x 0.25mm ID, 0.25µm or equivalent 5MS column
4.1.2 Fourier Transform–Infrared Spectrometer with Attenuated Total Reflectance (ATR) Accessory
4.1.3 Stereoscope
4.1.4 Polarizing Light Microscope
4.1.5 Top-loading Balance with Internal Calibration
4.1.6 Analytical Balance with Internal Calibration
4.1.7 Hotplate/Magnetic Stirrer
4.1.8 Electronic Rocker/Mixer, Speci-Mix
4.1.9 Refrigerator/Freezer (Frigidaire)
4.1.10 Refrigerator/Freezer (Haier)
4.1.11 Digital thermometers
4.1.12 NIST traceable thermometer
4.1.13 Adjustable pipettes (1 – 5 mL, 100 – 1000 µL, 20 – 200 µL, 5 – 50 µL, 0.5 – 10 µL)
4.1.14 Sonicator (Ultrasonic cleaner)
4.1.15 Certified weight sets (1mg – 500g, 1 kg, 5kg)

4.2 Supplies

4.2.1 Liquid Autosampler Glass Vials, 2 mL capacity screw or snap-on caps
4.2.2 Glass (clear and amber) Vials, screw caps
4.2.3 Spot plates
4.2.4 Disposable plastic transfer pipets, 3 mL capacity
4.2.5 Disposable glass transfer pipets with rubber bulbs
4.2.6 Disposable glass tubes
4.2.7 Glass (clear and amber) bottles with droppers

4.3 Performance Checks and Calibrations

Refer to Quality Manual, Section 2.5 and the Maintenance Plan SOP, QP102.5. Records of performance checks and calibrations are maintained according to Quality Manual, Section 1.13 and the Quality and Technical Records SOP, QP101.13.

4.4 Maintenance

Refer to Quality Manual, Section 2.5 and the Maintenance Plan SOP, QP102.5. Records of preventive and routine maintenance are maintained according to Quality Manual, Section 1.13 and the Quality and Technical Records SOP, QP101.13.

UNCONTROLLED COPY

5.0 Reagents, Standards and Controls

Refer to the Quality Manual, Section 2.1 and the Reagent Check SOP, QP102.1 for reagent check and labeling requirements.

5.1 Reagents

Chemicals and solvents used in reagents are of ACS reagent grade or higher. High quality, low residue solvents (e.g. HPLC grade, OMNISOLV, OPTIMA, or better) are preferred for instrumental analysis. Deionized (DI) or ultra-pure water is used to prepare reagents.

5.2 Reference Standards and Materials

5.2.1 Balance Weights

Class S-1, F or better weights are used to check the performance of the balances. Wear gloves when using the weights, either those provided by the manufacturer or those in the laboratory. Use the provided tweezers/forceps for smaller weights. Handle all weights with care since they can be easily damaged and smaller weights can be easily lost. The weights are stored in their original containers. When transporting, keep the weights in their original containers to prevent damage. The weights are calibrated tri-annually by an outside calibration laboratory.

5.2.2 Polystyrene Film

A Perkin Elmer Polystyrene film is provided to check the performance of the FT-IR Spectrophotometer.

5.2.3 Perfluorotributylamine (PFTBA)

PFTBA is used to calibrate the Gas Chromatograph-Mass Spectrometer (GC-MS). PFTBA is kept in a cylinder inside the GC/MS. Extra PFTBA solution is purchased from the manufacturer of the GC-MS and stored in the laboratory.

5.2.4 Drug Standards

All drug standards are entered into the Controlled Substances Inventory Log, TF202.5.1 by recording: date received, the receiver's initials, drug name, manufacturer, lot number, the observer's initials and amount received. When the standard is opened, the date opened and by whom is recorded.

When using any standard, the starting weight and ending weight are recorded on the Controlled Substances Usage Log, TF202.5.2. In addition the identity, lot #, usage date and initials of user and usage statement are recorded. To prevent contamination, only open one compound at a time and use clean labware and supplies. Wear appropriate gloves and

UNCONTROLLED COPY

other appropriate personal protective equipment (e.g. lab coat, safety glasses, and respirator) as needed for the compound. Refer to the MSDS for each compound of interest.

Standards are stored in their original containers. If a standard is transferred into a new container, make sure that the new container is clean and properly labeled to identify the contents. Use the same precautions to transport these substances as you would for handling evidence. When empty, the "date empty" is recorded on the appropriate sheet in the logbook and the bottle is discarded.

5.2.4.1 Primary Drug Standards

Primary standards are obtained from an approved vendor (List of Approved Vendors, QD009). Each new standard is run on GC-MS and FT-IR (as appropriate) prior to placement into service. Results are compared to known libraries and/or published spectra to confirm the identity of the standard. The standard is stored in the drug safe or locked evidence refrigerator and the paperwork generated from the analysis is filed in the Drug Standard Reference Binder.

If multiple vials with the same manufacturer and lot number are received, only one of the vials is tested by the aforementioned procedure. The vials are labeled A, B, C and so on as long as all of the vials have the same lot number.

5.2.4.2 Secondary Drug Standards

Secondary drug standards are obtained from previously analyzed case samples and laboratory synthesized samples. A Drug Acquisition Form, TF202.5.3 is used to document the acquisition and the transfer of material is witnessed by an authorized second party according to Purchasing SOP, QP101.6, Section 4.3. Secondary drug standards are entered into the Controlled Substances Inventory Log, TF202.5.1, by recording: date received, the receiver's initials, drug name, manufacturer, lot number, the observer's initials, amount received. The standard is stored in the drug safe or locked evidence refrigerator.

When using any secondary drug standard, the starting weight and ending weight is recorded on the Drug Acquisition.

5.2.4.3 Drug Standard Mixture

The drug standard mixture is prepared using the primary drug standards methamphetamine, MDMA, cocaine, methadone, heroin, and alprazolam with a concentration of less than 2 mg/ml. If a listed drug standard is not available it can be replaced with a similar compound. Drug standard mixture preparation is documented on the Drug Standard Preparation Log, TF202.5.4, to include the

UNCONTROLLED COPY

solvent system used. The drug standard mixture is run each week casework is performed and after significant maintenance.

5.2.5 Reference Materials

5.2.5.1 Spectral Libraries

The Crime Laboratory utilizes in-house, other laboratory and published spectral reference libraries (eg. IDDA, Clarke's, Forendex) for identification. The in-house spectral reference libraries are created by authorized laboratory personnel using verified compounds and methods appropriate to the analysis. They are maintained in electronic format, are easily accessible, and are updated as needed.

5.2.5.2 Training Materials

Secondary drug standards are used as training standards. The secondary standards are stored, handled, and transported similarly to the primary drug standards. Records are kept of their usage.

5.3 Controls

Appropriate controls are used throughout the analysis. For non-extracted samples analyzed on the GC-MS, a solvent blank is analyzed before each sample using the same parameters as the sample. For extractions analyzed on the GC-MS, an extraction blank (the extraction without the addition of a sample) is also analyzed on the same instrument and using the same parameters as the sample. For FT-IR, a background is collected prior to each sample analysis. Refer to the appropriate method for more specific information.

UNCONTROLLED COPY

6.0 Sample Management

6.1 Sample Management Procedure

6.1.1 Assigning a Laboratory Report Number (LR#)

All forensic drug samples are assigned a Laboratory Report number (LR#) according to Evidence Control SOP, QP102.8. Proficiency samples are treated exactly as case samples.

6.1.2 Entering Sample in Database

After a LR # is assigned to a sample, information about the sample is entered into the Sample Information Log according to Quality System Manual, QD001 and Evidence Control SOP, QP102.8. All examinations and procedures performed on each case sample are documented, saved and submitted for the case file. A Forensic Drug Analysis Case Notes form is used during the receiving and analysis process to document sample preparation, analysis and review.

6.1.3 Sampling

Sampling evidence is the most important initial step in forensic drug analysis. One must be sure that what is sampled is truly representative of the total population. The analyst must take into consideration the homogeneity (or lack thereof) among drug packaging (bags, packets, capsules, etc.) and its contents. Careful visual inspections and personal experience are essential in determining the proper sampling procedure. For items containing multiple specimens, statistically-based sampling models (e.g., hypergeometric distribution) allow the analyst to analyze a portion of the specimens and subsequently make statistical inferences about the population. Alternatively, a fixed number of specimens within a population may be analyzed with the purpose in mind of meeting the requirements of a particular criminal charge (e.g., simple possession, distribution). In these instances, an inference to the entire population will not be drawn and the number of specimens that were analyzed will be indicated on the report. Sampling plans are identified in the Sampling SOP, QP102.10.1.

6.2 Safety

Wear appropriate personal protective equipment (PPE) such as lab coats, gloves, and eye protection when working with unknown samples.

6.3 Departures from Methods and Procedures

The methods and procedures in this SOP are to be followed for all forensic drug analyses. If any departures are determined to be necessary, they must be reviewed, justified and approved by the Forensic Lab Manager.

6.4 Discovery Procedures

UNCONTROLLED COPY

All documentation is discoverable for a legal proceeding. Requests for documentation are to be made through the proper discovery outlets (e.g. the District Attorney's office). Documents are made available to the requesting party either by being provided to them or by request for appointment to review them on-site.

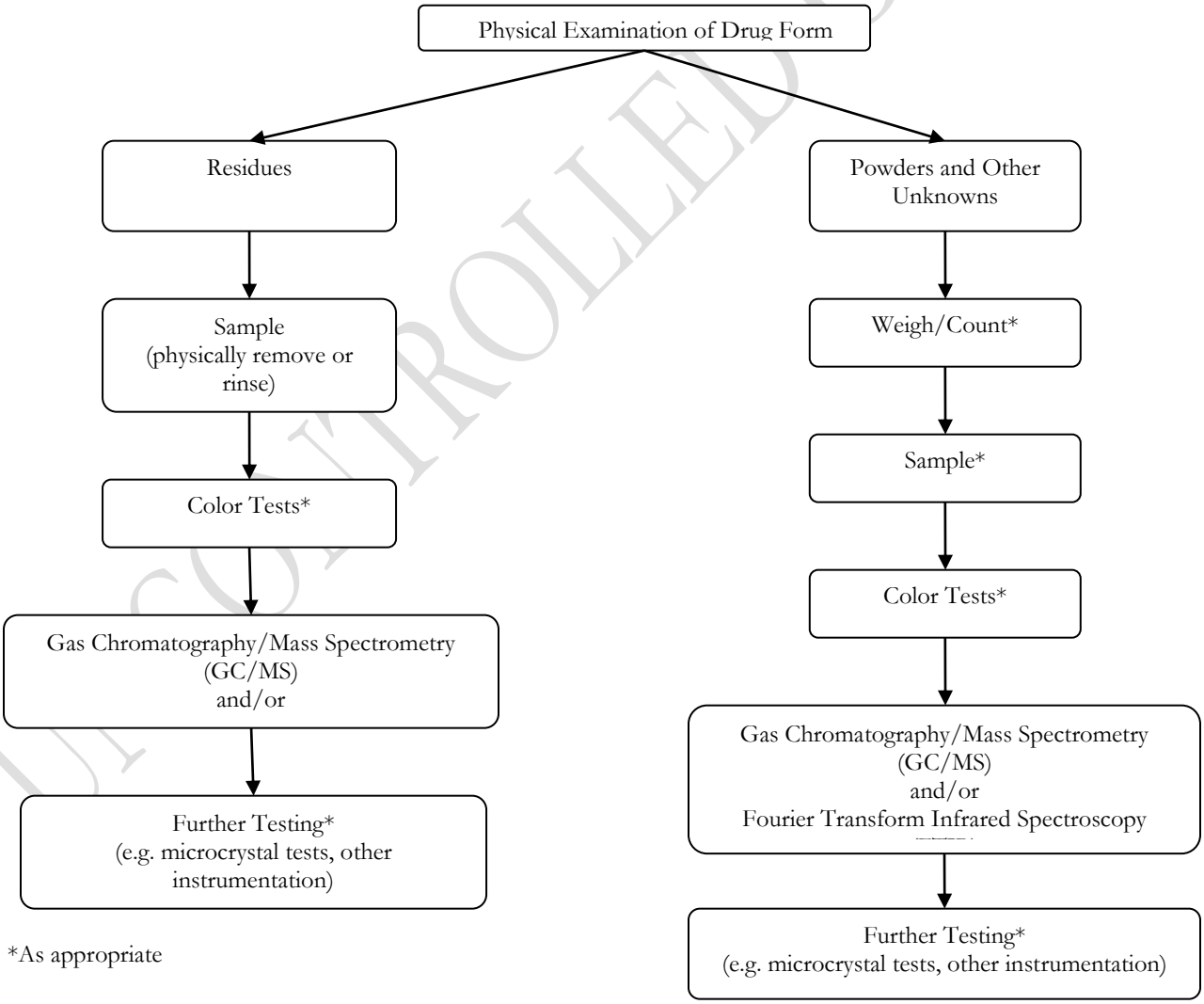
UNCONTROLLED COPY

7.0 Method

7.1 General Analytical Scheme

There are three general analytical schemes to be used for controlled substances. At various times, a forensic chemist will encounter drug substances for analysis that require specialized analysis. For these cases the flowchart for general unknowns can be followed and any modifications approved by the Forensic Lab Manager. It should be noted that sample size or other circumstances may require a rearrangement or modification of one or more steps. For these cases, the analyst may need to research and utilize resources outside of this general guide to determine the effective method for analysis. Any unusual methodologies must be approved by the Forensic Lab Manager.

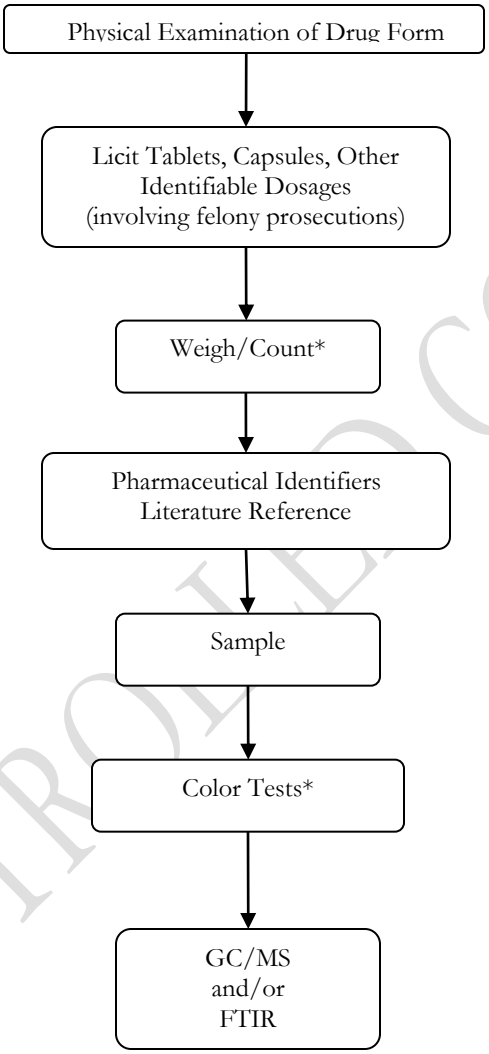
7.1.1 General Unknowns/Powders/Illicit Tablets



*As appropriate

UNCONTROLLED COPY

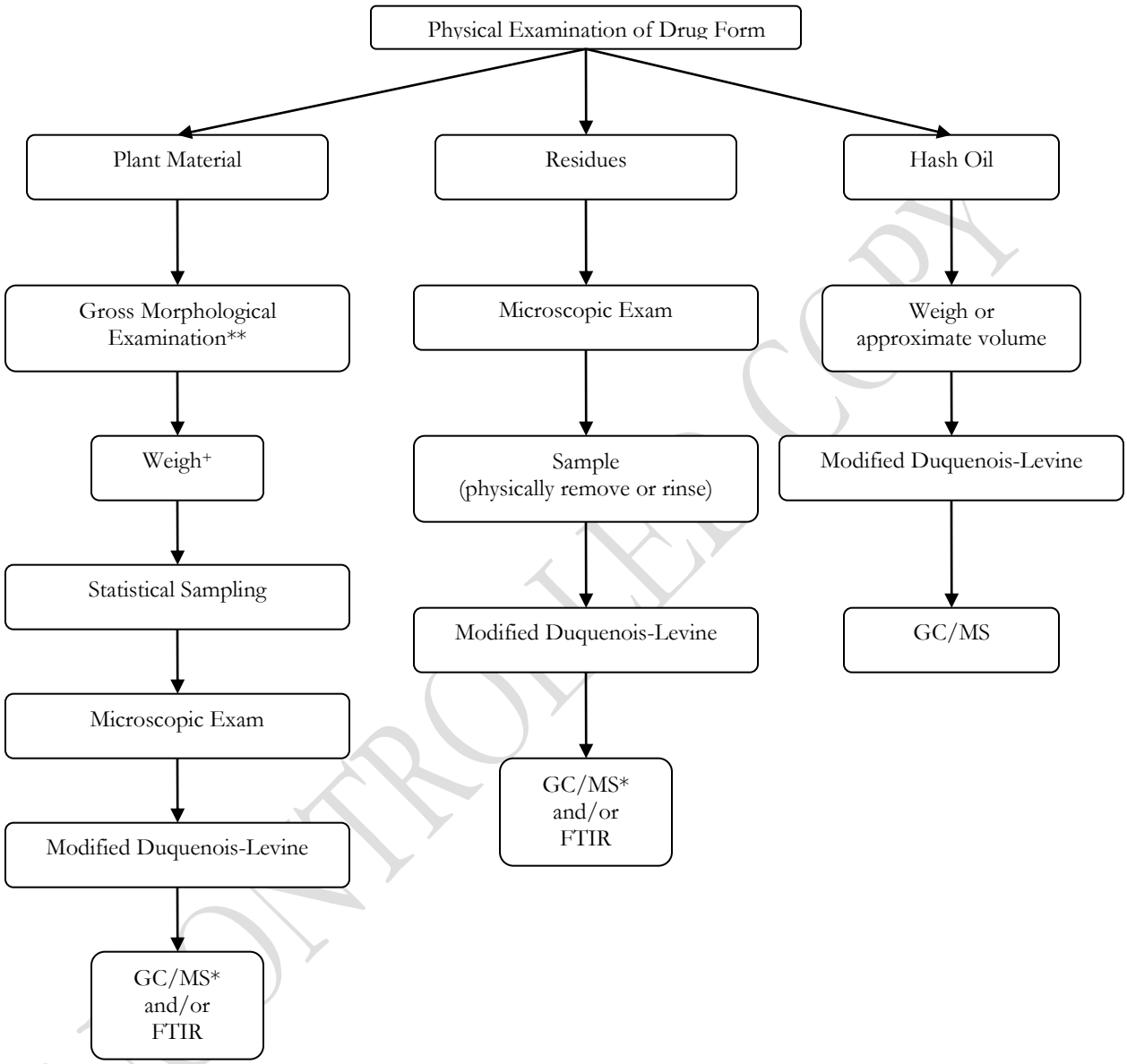
7.1.2 Tablets and Capsules



*As appropriate

UNCONTROLLED COPY

7.1.3 Marijuana



UNCONTROLLED COPY

+Gross weight suitable if less than ½ ounce w/innermost packaging
*Required if microscopic characteristics are absent or if another test is inconclusive
**As needed

7.1.4 Pharmaceutical Identifiers

7.1.4.1 Check an available reference, such as *Physician's Desk Reference*, Poison Control, *DEA Logo Index*, *Ident-a-drug*, *Drug ID Bible* or other similar sources, for information relating to inscriptions on tablets and capsules, size, color and shape.

7.1.4.2 If the drug is found to be a non-controlled prescription drug, no further testing is done. Two unrelated references are recommended for unfamiliar tablets if no further analysis will be conducted.

7.1.4.3 If the drug is found to be a controlled substance, it will be analyzed by GC/MS for authenticity.

7.1.4.4 For GC/MS analysis, the pill sample should be crushed (in a mortar and pestle) and diluted with suitable solvent. The excipients should be filtered and the filtered solution diluted with suitable solvent.

7.1.5 Color Tests

7.1.5.1 If the size of the sample is sufficient, perform the appropriate color tests required to provide an indication of any compounds present.

7.1.5.1.1 Take a small, representative sample of the suspected controlled substance to be placed on a clean spot plate or in a clean test tube.

7.1.5.1.2 To ensure a clean sampling well perform a blank test before addition of the sample. The color test reagent is added to the clean spot plate or test tube and observed for any color change.

7.1.5.1.2.1 If no color change is observed continue with the test.

7.1.5.1.2.2 If any color change is observed clean the sampling well or obtain a new sampling well and conduct the blank test again.

7.1.5.1.3 Add the sample to this unreacted reagent.

7.1.5.1.4 A change in the color of the reagent consistent with the color or color range specified in Appendix 15.1 for that specific color test is a positive result.

7.1.5.1.5 If after the addition of the sample in question the reagent does not change color or changes to a color that is not consistent with Appendix 15.1 for that specific color test then the result is negative.

UNCONTROLLED COPY

7.1.5.2 Refer to Appendix 15.1 for the interpretation of results. Refer to Color Test Working Instructions for reagent preparation and test specific procedures.

7.1.5.3 In order to discern subtle color changes a parallel blank analysis is conducted with each color test. This blank analysis follows the same procedure and acceptance criteria outlined above.

7.1.5.4 The analyst will perform a parallel check standard with secondary drug standards if the analyst is unfamiliar with the color test being performed or to confirm a color change. See Reagent Check SOP, QP102.1 for QC check standards of commonly used color test reagents. The check standard follows the same procedure and acceptance criteria stated above. If the check standard is negative refer to Reagent Check SOP, QP102.1.

7.1.6 Marijuana Analysis

7.1.6.1 Macroscopic Examination

7.1.6.1.1 Gross morphological characteristics that may be observed include the palmate arrangement of the leaflets, the pinnate appearance of the leaflets, the serrated edges of the leaflet, the buds (with or without seeds) and, if present, fluted stems and stalks.

7.1.6.1.2 Due to the compressed or mutilated nature of many samples, many of these characteristics may not be discernable.

7.1.6.1.3 Positive macroscopic examination results may be recorded in the analytical notes by the use of an abbreviation for positive and a short description of the characteristic(s) observed.

7.1.6.2 Microscopic Examination

7.1.6.2.1 View the sample at varying magnifications (approximately 10 – 40x) using a microscope.

7.1.6.2.2 Cystolithic hairs are unicellular, "bear claw" shaped hairs with a cystolith of $CaCO_3$ at the base. They are found in greatest abundance on the upper side of the leaf with longer covering hairs on the underside.

7.1.6.2.3 Seeds are coconut shaped, veined (with lacy markings) and have a ridge around the circumference.

7.1.6.2.4 The observation of the presence of appropriate cystolithic hairs or characteristic seeds is sufficient for a positive. The observation of

UNCONTROLLED COPY

additional characteristics is considered supportive. Positive microscopic examination results will be recorded in the analytical notes by the use of an abbreviation for positive by the appropriate characteristic.

7.1.6.3 Modified Duquenois-Levine Color Test

7.1.6.3.1 See section 7.1.5 for color test procedures and acceptance criteria.

7.1.6.3.2 Place approximately equal amounts of Duquenois reagent and concentrated HCl into a test well or test tube. Add small amount of sample. A positive reaction to the Duquenois portion is a blue/purple color.

7.1.6.3.3 Add sufficient $CHCl_3$ to form two discernable layers and mix. For a positive reaction to the Levine portion of the test, the bottom layer turns pink/purple in the presence of THC or other cannabinoids.

7.1.6.3.4 Record results in the case notes with an abbreviation for positive, an abbreviation for the colors of the each step may be noted as well. Negative reactions may be recorded in a similar fashion.

7.1.6.4 Gas Chromatography/Mass Spectrometry

GC/MS analysis is performed to confirm the presence of delta-9-THC. See section 7.1.7 for GC/MS procedures and acceptance criteria.

7.1.6.5 Acceptance Criteria

In order to confirm the presence of Marijuana in an unknown sample the analyst must find the presence of macroscopic and microscopic features of marijuana in addition to a positive Duquenois-Levine color test.

If the analyst cannot confirm the presence of macroscopic or microscopic features or if the Duquenois-Levine color test is negative the analyst must use additional testing, such as Gas Chromatography/Mass Spectrometry, to confirm the presence of delta-9-THC.

GC/MS confirmations of suspected marijuana samples are confirmed by laboratory personnel who are fully trained and authorized to perform all functions of the Forensic Drug Analysis program. A newly qualified forensic chemist will prepare suspected marijuana cases for GC/MS analysis for the first six (6) months of unsupervised casework; additional cases will be flagged by the laboratory director for GC/MS analysis as needed.

UNCONTROLLED COPY

7.1.7 Gas Chromatography/Mass Spectrometry

7.1.7.1 Dissolve the sample directly into a suitable solvent. If appropriate, extract the sample from an acidic or basic medium (or both if the contents of the sample are still unknown at this time).

7.1.71.1 Analyze a blank sample immediately before each case sample or quality control sample using the same parameters as the sample. The blank sample consists of the specific solvent or solvent system used to dissolve the case sample or quality control sample it accompanies.

7.1.7.1.2 For extractions analyzed on the GC-MS, an extraction blank (the extraction without the addition of a sample) is also analyzed on the same instrument and using the same parameters as the sample.

7.1.7.1.3 The GC results of the blank sample should be free of interfering peaks. Interfering peaks consist of those whose retention times would cause co-elution, poor resolution, or poor peak shape with the analyte(s) of interest. If interfering peaks are present flush the column with appropriate solvent until the interfering peaks are removed. If interfering peaks persist consult the Quality Manager for appropriate actions.

7.1.7.1.4 The MS results of the blank sample should be free of controlled substances. If controlled substances are present flush the column with appropriate solvent until the controlled substances are removed. If the controlled substances persist consult the Quality Manager for appropriate actions.

7.1.72 If the identity of the sample is unknown, the GC/MS provides further information, compare against a known standard.

7.1.7.3 A definitive structural identification technique such as MS or IR is required to be used on all substances where the identities are reported.

7.1.7.4 Acceptance Criteria

Integrated retention times for analytes agree with the standard within 2 seconds or 0.033 minutes for this to be considered a positive GC result. Standards are analyzed under the same conditions of sample for comparison.

In order for a mass spectrum to be considered definitive a molecular ion peak and two other major peaks must be present. The major peaks must have associated ^{13}C isotope peaks present.

UNCONTROLLED COPY

For compounds such as cocaine, heroin and LSD, a molecular ion peak with associated ^{13}C isotope peak must be present in order for the result to be considered definitive.

For compounds, such as methamphetamine, amphetamine and related compounds, it is imperative that the [M-H]+ ion and its associated ^{13}C isotope peak/molecular ion be present in mass spectra in order for the result to be considered definitive. (e.g. methamphetamine must have a 148 and 149 m/z ion)

For compounds that do not exhibit a molecular ion, examples include methylphenidate and fentanyl, the mass spectrum, when used in combination with retention time data and other testing, is sufficient for identification.

Refer to Appendix 15.3 for molecular ion peaks and major peaks of individual controlled substances.

When a known drug standard is not available in the laboratory standard spectral database, two external references must be used for identification. To be used, the reference spectra must be from two different sources or lot numbers of the standard and must meet the above acceptance criteria.

Compounds such as barbiturates and some benzodiazepines may need to be derivatized to improve chromatographic performance or confirm the predicted molecular ion. Techniques of derivatization include silylation, alkylation and acetylation.

For compounds identified and reported, anomalous mass peaks occurring above the molecular ion must be explained with data documentation in the case file. Easily recognizable column/septum bleed peaks, 207, 221, 267, 281, 327, 341, 355, 385, 415, and 429 m/z, occurring above the molecular ion may be labeled as such on the spectrum without further data documentation.

The strength of the sample/sensitivity of the instrument can be enhanced in the following ways, for example:

- Up to 4 uL of solution may be injected.
- The sample can be concentrated.
- The split can be lowered to 10:1.for split methods
- Splitless methods may be employed for samples containing small amounts of drugs including, for example, residues, LSD and fentanyl.

If the spectrum still does not meet the criteria, it is reported as "Insufficient for Identification".

7.1.8 Infrared Spectroscopy

UNCONTROLLED COPY

7.1.8.1 If the identity of the sample is unknown, the IR will provide further information.

7.1.8.1.1 A background sample is taken before all samples.

7.1.8.1.2 The background sample is reviewed for contamination; if none is present the sample is analyzed. If contamination is present the ATR sample window is thoroughly cleaned with suitable solvent and the background sample re-analyzed.

7.1.8.2 A definitive structural identification technique such as MS or IR is required to be used on all substances where the identities will be reported.

7.1.8.3 Acceptance Criteria

When using FTIR as the primary structural elucidation technique, the sample spectrum should compare favorably with a spectrum of a known standard in both its 1) overall appearance and 2) in the presence of at least six (6) or more major peaks in the fingerprint region (<1,700 cm^{-1}). Major peak ranges are listed in Appendix 15.2 for reference.

When a known drug standard is not available in the laboratory standard spectral database, two external references can be used for identification. To be used, the reference spectra must be from two different sources or lot numbers of the standard and must meet the above acceptance criteria.

Due caution should be exercised when using the similarity index generated by the library search algorithm. The library search is used as a tool to guide the analyst in the identification of minimum acceptance peaks.

When using FTIR to differentiate cocaine base from cocaine HCl or another salt form where GC/MS has been previously performed, the areas of the spectrum which are different between cocaine base and cocaine HCl should be clear. Other areas may have interfering peaks present that do not mask the "salt form" identity.

7.1.9 Further Testing

7.1.9.1 If the sample is still an unknown or other confirmation is needed, the analyst should use any instrumental techniques available (or combinations thereof) to arrive at a sound analytical conclusion about the identities of sample. This may involve using sources of instrumentation and techniques external to the Department such as DEA Special Testing or local Colleges and Universities (approval by the Forensic Lab Manager is required).

UNCONTROLLED COPY

7.1.9.2 Microcrystal tests are most often used for isomer determination only. They are to be used only in combination with a structural elucidation technique.

UNCONTROLLED COPY

8.0 Procedure

Technical procedures provided for forensic drug analysis are meant to guide the practices performed in the laboratory to ensure consistency is maintained throughout the forensic drug analysis process.

8.1 Evidence Control

All forensic drug evidence analyzed by the laboratory is received according to Quality Manual, Section 2.8 and Evidence Control SOP, QP102.8.

8.2 Evidence Examination

All forensic drug evidence is visually examined by the analyst once it is brought into the laboratory. The analyst confirms that the contents marked on the custody sheet and/or request form match the contents of the evidence package. Any discrepancies are noted in the analyst's case notes.

8.2.1 Evidence Discrepancies

Submitted forensic drug evidence is inventoried at the time of analysis. When significant discrepancies are discovered during inventory after comparing the submitted item(s) with the listed item(s) on the evidence tag/packaging, custody sheet and/or the Request for Physical Examination form, the Forensic Lab Manager is notified. A significant discrepancy may include but is not limited to overages or shortages in the number of evidence items submitted, items received that do not reconcile with the evidence description, and significant weight discrepancies. If there is a question as to whether a discrepancy exists, the Forensic Lab Manager is consulted.

When a significant discrepancy is discovered, the Forensic Lab Manager verifies the discrepancy at or near the time it is discovered and initials and dates the case notes. If deemed necessary, the Forensic Lab Manager consults the Office of the Chief, Professional Standards Division.

In the vast majority of forensic drug cases, the weights listed on an evidence tag/packaging, custody sheet and/or the Request for Physical Examination form by an agency are gross weights. The packaging for forensic drug evidence varies greatly; therefore, the gross weights may be significantly different than the net weights obtained during analysis. It is not possible to quantify a weight discrepancy that warrants notifying the supervisor because of the variability in the packaging of drug evidence, the unknown condition of the balances used, the procedure individual officers follow to determine the weight of forensic drug evidence, and other variables that may be involved in determining weights of drug evidence.

If in the trained analyst's opinion a significant discrepancy exists, the Forensic Lab Manager is notified so appropriate action can be taken to resolve the issue. It is always best to err on the side of caution when making a decision whether a discrepancy exists.

UNCONTROLLED COPY

The Forensic Lab Manager determines the significance of the discrepancy and takes appropriate action as necessary. If it is determined that there is a potential problem, necessary personnel are notified and proper notation made in the case file.

8.3 Sampling

Sampling plans are identified in the Sampling SOP, QP102.10.1 and are followed for all casework.

8.4 General Analytical Procedure

8.4.1 Introduction

The correct identification of a drug or chemical depends on the use of an analytical scheme based on validated methods and the competence of the analyst. It is expected that, in the absence of unforeseen circumstances, an appropriate analytical scheme effectively results in no uncertainty in reported identifications. Multiple uncorrelated techniques are used.

8.4.2 Categorizing Analytical Techniques

Techniques for the analysis of drug samples are classified into three categories based on their maximum potential discriminating power. However, the classification of a technique may be lower, if the sample, analyte or mode of operation diminishes its discriminating power.

Examples of diminished discriminating power may include:

- an infrared spectroscopy technique applied to a mixture which produces a combined spectrum
- a mass spectrometry technique which only produces molecular weight information

8.4.2.1 Categories of Analytical Techniques

Category A - Infrared Spectroscopy, Mass Spectrometry, Nuclear Magnetic Resonance, Raman Spectroscopy, X-ray Diffractometry

Category B – Capillary Electrophoresis, Gas Chromatography, Ion Mobility Spectrometry, Liquid Chromatography, Microcrystalline Tests, Thin Layer Chromatography, Pharmaceutical Identifiers, Cannabis only: Macroscopic Examination and Microscopic Examination

Category C – Color Tests, Fluorescence Spectroscopy, Immunoassay, Melting Point, Ultraviolet Spectroscopy

UNCONTROLLED COPY

8.4.2.2 The following minimum standards must be met in order for an identification of a drug or chemical to be reported.

8.4.2.2.1 When a validated Category A technique is incorporated into an analytical scheme, at least one other technique (from either Category A, B or C) shall be used.

8.4.2.2.2 When a Category A technique is not used, at least three different techniques shall be employed. Two of the three techniques shall be based on uncorrelated techniques from Category B.

8.4.2.2.3 For cannabis, macroscopic and microscopic examinations will be considered as uncorrelated techniques from Category B when observations include documented details of botanical features. Along with presence of leaves, stems, seeds, and hairs there must be the presence of cystolithic hairs at a minimum to meet acceptance criteria that the plant material is cannabis.

8.4.2.2.3.1 For exhibits of cannabis that lack sufficient observable macroscopic and microscopic botanical detail (e.g. extracts or residues), 9-tetrahydrocannabinol (THC) or other cannabinoids shall be identified utilizing the principles set forth in sections 8.4.2.2.1 and 8.4.2.2.2.

8.4.3 Drug Identification Criteria

8.4.3.1 In cases where hyphenated techniques are used (e.g. gas chromatography-mass spectrometry), they are considered separate techniques provided that the results from each both meet the acceptance criteria.

8.4.3.2 The chosen analytical scheme demonstrates the identity of the specific drug present and shall preclude a false positive identification and minimize false negatives. Where a scheme has limitations, this shall be reflected in the final reported identification.

8.4.3.3 These are minimum standards for the forensic identification of commonly seized drugs. However, it should be recognized that they may not be sufficient for the identification of all drugs in all circumstances. Within these recommendations, it is up to the analyst to determine which combination of analytical techniques is used to identify a controlled substance.

8.4.3.4 For most cases, perform a color test, analysis by FT-IR and analysis by GC/MS.

UNCONTROLLED COPY

9.0 Parameters

9.1 Trace Ultra Gas Chromatograph Operational Parameters

The following parameters are recommended as starting parameters. Parameters may need to be adjusted to maximize performance.

Trace Ultra Gas Chromatograph	
Oven temperature range:	70 – 300 °C
Temperature ramp:	20 °C/min
Hold time:	9 min
Inlet temperature:	200 °C
Split/Splitless mode:	split
Split ratio:	100:1
Column flow:	1 mL/min
Transfer line temperature:	300 °C

9.2 DSQ II Mass Spectrometer Operational Parameters

The following parameters are recommended as starting parameters. Parameters may be adjusted to maximize performance.

DSQ II Mass Spectrometer	
Source temperature:	250 °C
Detector gain:	1.0×10^5
Multiplier voltage:	1111 V
Scan mode:	Full scan
Scan time:	0.25 sec
Scan range:	40 – 450

9.3 Spectrum 100 Fourier Transform Infrared Spectrometer Operational Parameters

The following parameters are recommended as starting parameters. Parameters may be adjusted to maximize performance.

Spectrum 100 FT-Infrared Spectrometer	
Scan range:	4000 – 550 cm^{-1}
Scan number:	8
Resolution:	4.0 cm^{-1}

UNCONTROLLED COPY

10.0 Quality Control

10.1 Introduction

The purpose of this section is to provide a uniform Quality Assurance Program for the Forensic Drug Analysis. It is to establish a baseline or reference point of reliability and system performance.

It is expected that the analyst will report any unacceptable or anomalous behavior of any of our analytical systems to the Quality Manager to be handled according to the Control of Nonconforming Work SOP, QP101.9.

10.2 Reagents

10.2.1 Chemicals and solvents used in qualitative reagents should be of at least ACS reagent grade.

10.2.2 Preferred solvents used to dissolve samples or standards are high quality, low residue solvents (e.g., HPLC grade, OMNISOLV, OPTIMA).

10.2.3 Water used in reagent preparation should be either deionized (DI) or ultrapure.

10.2.4 Stock solutions of general color test reagents are made up as needed and reagent preparation recorded on the Drug Reagent Preparation Log, QF202.1.1. Follow the Reagent Check SOP, QP102.1.

10.2.5 Color test reagents are verified every three months per Reagent Check SOP, QP102.1 and check recorded on the Drug Reagent QC Check Log, QF202.1.2.

10.2.6 Analysts must document the use of unique reagents other than those in Reagent Check SOP, QP102.1, Appendix A, Common Reagents and Quality Control Standards, and are responsible to check them with an appropriate standard and record on the Drug Reagent Preparation Log, QF202.1.1. Refer to the Color Tests Working Instructions for guidance on preparing and checking color test solutions.

10.3 Standards

10.3.1 Standards used as reference materials in casework are considered critical supplies and are purchased from the List of Approved Vendors, QD009.

10.3.2 For all standards obtained for drug lab use, a qualified analyst obtains a mass spectrum and/or IR prior to entry into the laboratory database library.

10.3.3 The analyst gathers the data and confirms the identity of the standard using the acceptance criteria from Sections 7.1.6.4 and 7.1.7.3. Identify on the Controlled Substances Usage Log the analytical method(s) used to confirm the standard. The

UNCONTROLLED COPY

obtained chromatogram and spectra are printed, initialed and dated by the analyst and stored in the Drug Standard Reference binder for future use.

10.3.4 If an analyst needs a standard from a new lot that has not been documented in this fashion, the analyst performs the above procedure prior to using it for drug case work.

10.4 Refrigerators and Freezers

10.4.1 The temperature of refrigerators and freezers which store reagents, standards or evidentiary material are checked and recorded on a weekly basis using the Refrigerator and Freezer Temperature Log, TF201.17.

10.4.2 For refrigerators, the temperature shall be between 2 – 8 ° C.

10.4.3 For freezers, the temperature shall be below -20 ° C.

10.4.4 If temperatures fall outside the range, the thermostat is adjusted. If necessary, the contents of the refrigerator or freezer are moved to another refrigerator or freezer temporarily and the new location's temperature is monitored

10.4.5 Critical reagents and standards should be re-verified if the temperature in the refrigerator exceeds 15 ° C or the freezer exceeds 0 ° C prior to use in case work.

10.5 Balances

10.5.1 Daily Performance Check

Each day a balance is used it is verified using one mid-range NIST traceable weight standard.

10.5.2.1 Follow the procedure outlined in 10.5.2 using only one mid-range NIST traceable weight standard. Refer to Table 10.5 for appropriate check weights and specific acceptance criteria.

10.5.2.2 If a result from the performance check is outside of the acceptable range, first ensure that the balance is level and clean prior to rechecking.

10.5.2.3 If applicable, use the internal calibration function of the balance prior to rechecking.

10.5.2.4 If a result is outside of the acceptable range after performing the actions found in 10.5.2.2 and 10.5.2.3, the balance is immediately taken out of service until maintenance and/or calibration are performed by an approved vendor and the Quality Manager is notified.

UNCONTROLLED COPY

All daily performance checks are recorded in the Daily Balance Performance Check Log, TF202.10.4 Logs are kept in the Diluter and Balance binder in the laboratory until they are archived electronically. Completed pages of the log are maintained per the Quality and Technical Records SOP, QP101.13.

Table 10.5 Balances and Appropriate Check Weights

Balance Type	**Balance Example**	**Check Weights**
Analytical Balance	Mettler Toledo XS603S	0.100 g (± 0.002 g) 1.000 g (± 0.020 g) 10.000 g (± 0.200 g) 100.000 g (± 2.000 g) 500.000 g (± 10.000 g)
Top-loading Balance	Denver SI-4002	1.00 g (± 0.02 g) 10.00 g (± 0.20 g) 100.00 g (± 2.00 g) 500.00 g (± 10.00 g) 1000.00 g (± 20.00 g)
High Capacity	Ohaus T5IP	0.50 kg (± 0.01 kg) 1.00 kg (± 0.02 kg) 5.00 kg (± 0.100 kg)

10.5.2 Weekly Performance Check

All analytical and top-loading balances are checked each week prior to using for casework Class S-1 weights or better. All high capacity balances are checked prior to use with Class S-1 or Class F weights or better. Record the weights on the Balance Performance Check Log, TF202.10.1, date and initial. Refer to Table 10.5 for appropriate check weights and specific acceptance criteria.

10.5.2.1 Tare balance and place weight on it.

10.5.2.2 Record the weight and confirm it is ±2 % of the known weight value.

10.5.2.3 If a result from the performance check is outside of the acceptable range, first ensure that the balance is level and clean prior to rechecking.

10.5.2.4 If applicable, use the internal calibration function of the balance prior to rechecking.

10.5.2.5 If a result is outside of the acceptable range after performing the actions found in 10.4.1.3 and 10.4.1.4, the balance is immediately taken out of service until maintenance and/or calibration are performed by an approved vendor.

UNCONTROLLED COPY

10.5.3 Accuracy and precision are established prior to placing a balance into service after purchase or repair.

10.5.3.1 The Balance Accuracy and Precision Check worksheet, TF202.10.2 is used for this purpose.

10.5.3.2 The check weights are weighed and recorded five times.

10.5.3.3 The mean and % relative standard deviation (%RSD) are calculated for each weight.

10.5.3.4 The accuracy of each weight must be within ±2 % of the known weight value.

10.5.3.5 %RSD must be less than or equal to 5 %.

10.5.3.6 The balance is immediately taken out of service if these criteria are not met and the nonconforming work is reported to the Quality Manager and is addressed using the Nonconforming Work SOP, QP101.9

10.5.4 Calibrations/Certifications

10.5.4.1 Balances are calibrated by an outside vendor annually.

10.5.4.2 Weights used to check balance accuracy are sent to vendor for re-certification every three years.

10.6 Gas Chromatograph/Mass Spectrometer (GC/MS)

10.6.1 Record any maintenance performed in the Maintenance Log, date and initial.

10.6.2 Daily

A maintenance tune is performed prior to any drug case work analysis each day. A qualified analyst verifies the maintenance tune against the current autotune and initials and dates the tune report prior to use of GC/MS for casework. The maintenance tune report is saved electronically on the GC/MS computer and the initialed tune report is scanned and stored electronically per the Quality and Technical Records SOP, QP101.13. If the maintenance tune fails, check PFTBA calibration gas and perform maintenance. If maintenance tune continues to fail contact the service engineer and notify the Quality Manager.

Check PFTBA calibration gas and add as needed.

10.6.3 Weekly

UNCONTROLLED COPY

An autotune is performed prior to any drug case work analysis each week. Refer to Table 10.6 for autotune acceptance criteria. If the autotune fails check PFTBA calibration gas and perform maintenance. If autotune continues to fail contact the service engineer and notify the Quality Manager. A qualified analyst verifies the autotune meets acceptance criteria and initials and dates to confirm verification. The autotune report is saved electronically on the GC/MS computer and the initialed tune report is scanned and stored electronically per the Quality and Technical Records SOP, QP101.13.

Table 10.6 Autotune Acceptance Criteria

Tune Parameter	**Specific Parameter**	**Acceptance Range**
Peak Intensity	Peak Intensity of 69	>10,000,000
% Base Peak	% Base Peak of 219	>40%
% Base Peak	% Base Peak of 414	>2%
% Base Peak	% Base Peak of 502	>2%
RF Frequency		<2000 kHz
Detector	3e5	≤ 2200 V
Leak Check	% of Reference	<5%
Prefilter Offset		>-11 V
Force Pressure		≤ 60 mTorr

The column performance is checked by injecting the drug standard mixture prior to using it each week of drug case work. Run the mixture using an appropriate general drug screen method. The data is saved electronically on the GC/MS computer. The drug standard mixture chromatogram is printed and verified by a qualified analyst before storing in the Drug Standard and Reagent binder.

The chromatogram should demonstrate good chromatographic performance through peak resolution and peak shape of controlled substances. The mass spectra should correctly identify all controlled substances and meet the acceptance criteria outlined in Section 7.1.6.4. If the above criteria are not met the analyst conducts troubleshooting that may include method modification, GC maintenance, autotune, source cleaning, or manufacturer's service.

10.6.3 Any performance discrepancies or degradation is reported to the Quality Manager.

10.6.4 After significant maintenance has been performed, autotune the instrument and run the drug standard mixture.

10.7 Fourier Transform Infrared Spectrophotometer (FTIR)

10.7.1 Record any maintenance performed in the Maintenance Log, date and initial.

10.7.2 Daily

UNCONTROLLED COPY

A system suitability check, to include a contamination check, is performed prior to any drug case work analysis each day. Refer to Table 10.7 for system suitability acceptance criteria. A qualified analyst verifies the system suitability meets acceptance criteria and initials and dates to confirm verification. The system suitability report is saved electronically on the FTIR computer and the initialed report is scanned and stored electronically per the Quality and Technical Records SOP, QP101.13. If system suitability fails clean the ATR window and retry. If check continues to fail contact the service engineer and notify the Quality Manager.

Table 10.7 System Suitability Acceptance Criteria

System Suitability Criteria	Specific Parameter	Acceptance Criteria
Abscissa Check	Peak 3060.00 cm^{-1}	3058.00 – 3062.00 cm^{-1}
	Peak 1601.00 cm^{-1}	1598.00 – 1603.00 cm^{-1}
	Peak 1028.00 cm^{-1}	1025.00 – 1030.00 cm^{-1}
Noise Check	RMS (%T)	< 0.0824 %T
	Peak to Peak (%T)	< 0.4605 %T
	Trend (%)	< 0.0442 %T
Throughput Check	4000.00 cm^{-1} (%T)	> 80.00 %T
	2600.00 cm^{-1} (%T)	> 80.00 %T
	1000.00 cm^{-1} (%T)	> 80.00 %T
Contamination Check	3100.00 – 2800.00 cm^{-1} (A)	< 0.100 A
	1800.00 – 1600.00 cm^{-1} (A)	< 0.100 A
	1400.00 – 1100.00 cm^{-1} (A)	< 0.100 A

10.7.3 Weekly

A standard of procaine hydrochloride (HCl) is used as a QC check. Refer to Section 7.1.7.3 for acceptance criteria. If the acceptance criteria are not met, check for contamination and re-analyze. If the QC check continues to fail, contact the service engineer and notify the Quality Manager. A qualified analyst verifies the QC check meets acceptance criteria and initials and dates and stores in the Drug Standard and Reagent binder. The weekly QC check spectrum is saved electronically on the FTIR computer.

10.7.4 After significant maintenance has been performed, run the daily and weekly QC checks.

10.8 Proficiency Testing

Proficiency testing is required at least once per year per analyst to show that the methods and procedures for forensic drug analysis produce valid and accurate results. Proficiency tests meet the criteria in Quality System Manual, QD001, Section 2.9. Records of all proficiency testing are maintained per the Quality and Technical Records SOP, QP101.13.

UNCONTROLLED COPY

10.9 Non-conforming Work

When non-conforming work is identified (e.g. repeated blank or QC failures) the analyst stops work and reports the nonconforming work to the Quality Manager. The nonconforming work is addressed using the Nonconforming Work SOP, QP101.9.

10.10 Measurement Uncertainty

Measurement uncertainty is estimated for forensic drug analysis using the simplified Guideline to Uncertainty Measurement (GUM) eight step approach. These steps are outlined in the following paragraphs. The measurement uncertainty estimate is reviewed on an annual basis when the balance is recalibrated or more often as deemed necessary by the Quality Manager. The Measurement Uncertainty Form, TF 202.10.5, is used to document each measurement uncertainty estimate performed. Any calculations or data used for the estimate not documented on the form are included as attachments.

The coverage probability used for measurement uncertainty estimates is 99.8%. The measurement uncertainty estimate is reported to only two decimal places and the results of any calculations will be rounded up, in order to maintain a conservative estimate. During estimation ensure all units are the same for calculations, data can be converted to percentage to simplify calculations.

10.10.1 Simplified GUM Approach

First step is to define what is being measured, which can be done with a short statement or a quantitative expression. Figure 10.5.5 gives an example of the Measurement Uncertainty Form, TF 202.10.5, completed with all eight steps.

The second step is to identify potential sources of uncertainty that contribute to the final result's uncertainty. This can be done in a list format or a graphical format. An example of a graphical format for forensic drug analysis is shown in Figure 10.5.1.

UNCONTROLLED COPY

Step 2: Cause & Effect Diagram

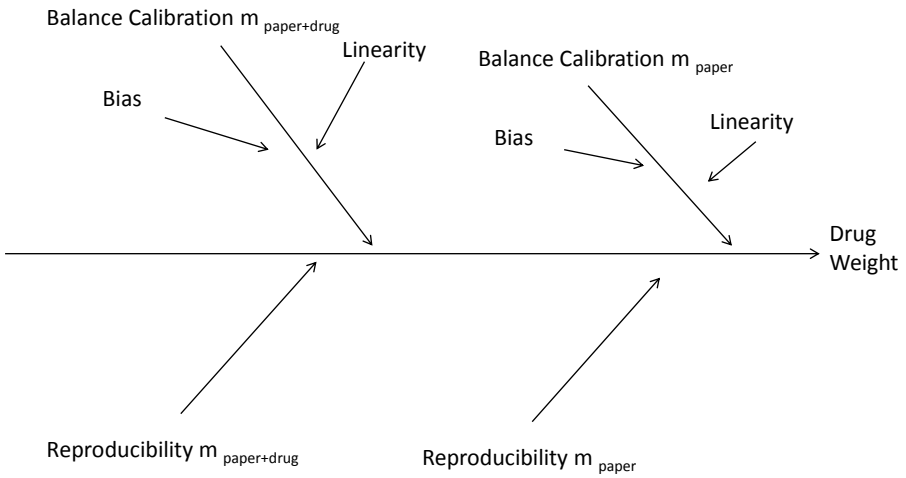


Figure 10.10.1 Potential Sources of Uncertainty

The third step is to reconcile these sources of uncertainty to see if any are adequately accounted for with existing data and can therefore be removed from consideration. Figure 10.10.2 shows how these data can be reconciled for forensic drug analysis.

UNCONTROLLED

Step 3: Reconcile Contributors

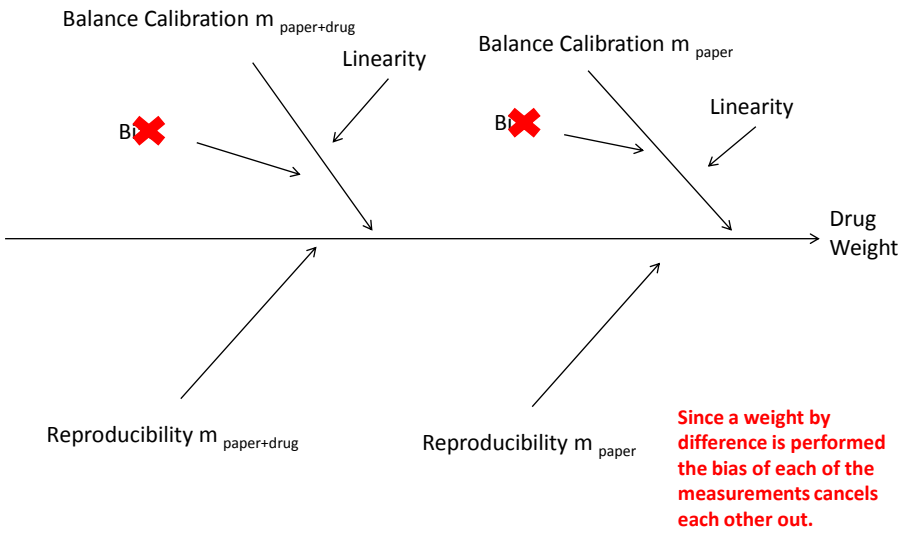


Figure 10.10.2 Reconcile Sources of Uncertainty

The fourth step is to quantify the sources of uncertainty. Figure 10.10.3 shows the remaining sources of uncertainty that affect the final uncertainty of the result. The analyst performing the estimate determines if data already exists for all sources or if further research or experimentation is required.

UNCONTROLLED

Step 4: Quantify Uncertainty Sources

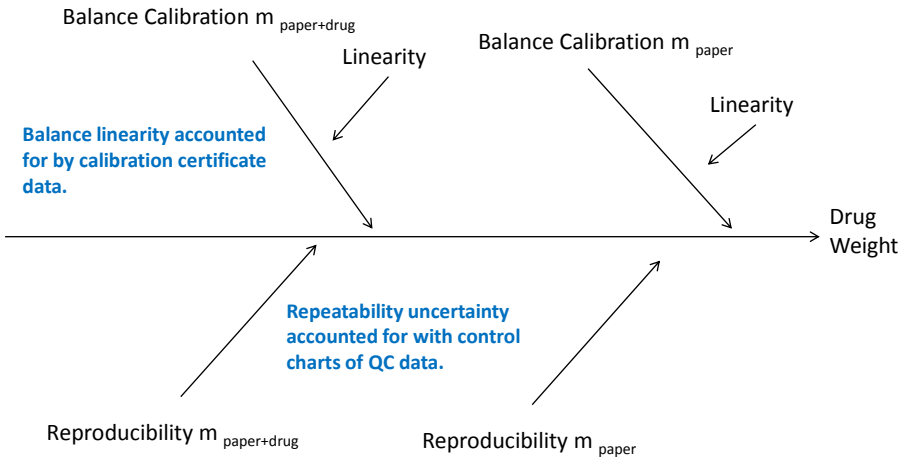


Figure 10.10.3 Remaining Sources of Uncertainty

In addition to determining a value for the uncertainties the characteristics of the data used must be identified as well. The data is categorized as Type A or Type B error and the distribution model is identified.

Type A data is derived from multiple measurements conducted over a period of time and statistically analyzed, this data usually comes from control charts of QC data or validation studies. If data does not exist for a new or unique method then experimentation is performed to obtain reproducibility data. Type B data is not measured statistically by the lab, is usually considered individually, and can be minimized through optimization; type B data usually consists of instrument or standards' calibration performed by an outside source.

The distribution model of the data must be identified so the proper divisor to obtain one standard uncertainty, or 1σ, can be used. The distribution of the data is typically normal, also known as Gaussian, student t, or rectangular. Most calibration certificate data can be assumed to be normal and the divisor derived from the confidence level (ie. 95% = 2σ, so divisor is 2). Control chart or validation data may be normal or a student t distribution, with the former requiring 100 data points, but typically has a divisor of 1. The student t table, Figure 10.5.4, is consulted to expand the uncertainty when the largest uncertainty contributor is of this distribution. If the distribution of the data is unknown it can be considered rectangular with a divisor of square root of three.

The fifth step is to perform the calculation to obtain the standard uncertainties.

UNCONTROLLED

In the sixth step the standard uncertainties are combined. If any standard uncertainty is less than one third of the highest uncertainty then it can be removed due to it having such a small effect on the final uncertainty. The remaining standard uncertainties are reviewed to determine if any are correlated, if they are then they can be cancelled out, but typically at this point all uncertainties are uncorrelated. All uncorrelated uncertainties are combined using the root sum of squares equation.

The seventh step is to express the expanded uncertainty. Based on the confidence level of 99.8% the coverage factor is determined using either the normal distribution value (k=3) or the student t table, Figure 10.10.4, value. The combined uncertainty is then multiplied by the coverage factor to determine the expanded uncertainty. Figure 10.10.5 shows an example of the Measurement Uncertainty Form, TF 202.10.5, completed.

Once the analyst has completed the calculation, the Measurement Uncertainty Form along with any attachments is reviewed by the Quality Manager for approval.

n-1	95% Confidence Level	99.8% Confidence Level	n-1	95% Confidence Level	99.8% Confidence Level
1	12.7	318.3	16	2.1	3.7
2	4.3	22.3	17	2.1	3.6
3	3.2	10.2	18	2.1	3.6
4	2.8	7.2	19	2.1	3.6
5	2.6	5.9	20	2.1	3.6
6	2.5	5.2	30	2.0	3.4
7	2.4	4.8	40	2.0	3.3
8	2.3	4.5	50	2.0	3.3
9	2.3	4.3	60	2.0	3.2
10	2.2	4.1	70	2.0	3.2
11	2.2	4.0	80	2.0	3.2
12	2.2	3.9	90	2.0	3.2
13	2.2	3.9	100	2.0	3.2
14	2.1	3.8	∞	2.0	3.1
15	2.1	3.7			

Figure 10.10.4 Student t Table

UNCONTROLLED COPY

MEASUREMENT UNCERTAINTY DETERMINATION FORM

Measurand Statement (what is the quantity being measured)

Weight Reporting of a Drug (mass of drug = mass of paper + drug - mass of paper)

Traceability Statement (how it is established for the measurement)

Traceability is established through external calibration of all instruments used and monitoring quality control data using control charts.

Technical Procedure (w/ Section, if applicable) TP102, FDA SOP	**Analyst** A. Hutson	**Date** 12/11/2013

Equipment Used for Measurement (list all equipment considered in uncertainty estimation)

Mettler Toledo XS603SDR balance

Sources of Uncertainty	Type A or Type B?	Std Dev (units)	Distribution Model	Divisor	Std Uncertainty (1σ)	Significant (Y/N)	Uncertainty Data From?
Repeatability m p	A	0.001 g	student t	1	0.001 g	Y	Control charts
Repeatability m p+d	A	0.001 g	student t	1	0.001 g	Y	Control charts
Linearity m p	B	0.008 g	normal	2	0.004 g	Y	Balance cal cert
Linearity m p+d	B	0.008 g	normal	2	0.004 g	Y	Balance cal cert

Equation for Combined Uncertainty ($U_{combined}$) = sqrt {(0.001)^2 + (0.001)^2 +(0.004)^2 + (0.004)^2} = 0.006

Combined Uncertainty ($U_{combined}$)	0.006 g		
Confidence Level	99.8%	**Coverage Factor (k)**	3
Expanded Uncertainty (=$U_{combined}$ * k)	= 0.006*3 = 0.018, rounded up to 0.020 g		
Date for Review 8/31/2014	**Quality Manager Approval**		

Figure 10.10.5 Example Measurement Uncertainty Form, TF 202.10.5

10.10.2 Reporting Uncertainty

The last step of the simplified GUM approach is reporting uncertainty with the final result. The uncertainty is reported to the same significance as the measured value, is always rounded up during the expanded uncertainty calculation, and has the same units and precision of the final result. A statement of the confidence level of 99.8% is included when reporting the uncertainty with the final result.

When individual weights are added to calculate a total combined weight, the uncertainties associated with each individual value must be taken into account in the total uncertainty. The Root Sum Square method is used for calculation.

$$\text{Total combined uncertainty} = \sqrt{(U_{combined} * k)^2 * n}$$

For example, if five items are weighed, each with an uncertainty of 0.020 grams, then the reported uncertainty is:

$$\text{Total combined uncertainty (5 bindles)} = \sqrt{(0.020)^2 * 5} = 0.045$$

The uncertainty in the example above is calculated in the units of grams which are the same units for the reporting weight. The final result will be in the format x ± y at 99.8% confidence, where x is the result and y is the uncertainty. Forensic drug analysis results will be reported out to the number of decimal places on the balance used with the uncertainty to the same significance and a statement of the confidence level. An example of a reported result is "1.234 ± 0.020 grams at 99.8% confidence.

Refer to Measurement Traceability and Uncertainty SOP, QP102.4.6.
Refer to Measurement Uncertainty Form, TF 202.10.5.
Refer to Reporting Results SOP, QP102.10.

UNCONTROLLED COPY

11.0 Calculations

11.1 Balance Accuracy and Precision Check

Accuracy and precision are established prior to placing a balance into service after purchase or repair.

11.1.1 The Balance Accuracy and Precision Check worksheet, TF202.10.2 is used for this purpose.

11.1.2 The check weights are weighed and recorded five times.

11.1.3 The mean and % relative standard deviation (%RSD) are calculated for each weight.

$$\%RSD = 100 * (\text{standard deviation} / \text{mean})$$

11.1.4 The accuracy of each weight must be within ±2 % of the known weight value.

11.1.5 %RSD must be less than or equal to 5 %.

UNCONTROLLED COPY

12.0 Reporting

12.1 Reviewing the Results

Prior to reporting results of an analysis, the analyst will:

1) Review the results of each analytical method used verifying that acceptance criteria detailed in Section 7 have been met.
2) Initial and date each page in the case notes and all data sheets.
3) Review category A, B and C results to confirm results are in agreement.
4) Ensure that the following criteria from Section 8.4 are met for an identification to be reported:

    a. When a validated Category A technique is incorporated into an analytical scheme, at least one other technique (from either Category A, B or C) shall be used.

    b. When a Category A technique is not used, at least three different techniques shall be employed. Two of the three techniques shall be based on uncorrelated techniques from Category B.

    c. For cannabis, macroscopic and microscopic examinations will be considered as uncorrelated techniques from Category B when observations include documented details of botanical features. Along with presence of leaves, stems, seeds, and hairs there must be the presence of cystolithic hairs at a minimum to meet acceptance criteria that the plant material is cannabis.

        1) For exhibits of cannabis that lack sufficient observable macroscopic and microscopic botanical detail (e.g. extracts or residues), 9-tetrahydrocannabinol (THC) or other cannabinoids shall be identified utilizing the principles set forth in sections 8.4.2.2.1 and 8.4.2.2.2.

12.2 Preparing the Report

A report is created using the Laboratory Report Template – Forensic Drug Analysis, QD006. The analyst fills out all pertinent information from the Sample Information Log. Any information that is not applicable is labeled as N/A.

The results are reported in accordance with the reporting requirements in Section 4.9 of the Sampling SOP, QP102.10.1. Refer to Reporting Results SOP, QP102.10, for specific information on reporting.

If a controlled substance is not detected, the result is reported as "No controlled substance detected." When possible, indicate in the case notes the suspected identity of the uncontrolled substance.

UNCONTROLLED COPY

12.3 Technical Review of the Results

Technical review of a run is performed by someone trained and authorized to perform the review other than the analyst who performed the analysis. The technical reviewer will:

1) Review the results of each analytical method to ensure the appropriate method was used verifying that acceptance criteria detailed in Section 7 are met .
2) Initial and date each page in the case notes and all data sheets after review.
3) Review category A, B and C results to confirm results are in agreement.
4) Ensure that the following criteria from Section 8.4 are met:

    a. When a validated Category A technique is incorporated into an analytical scheme, at least one other technique (from either Category A, B or C) shall be used.

    b. When a Category A technique is not used, at least three different techniques shall be employed. Two of the three techniques shall be based on uncorrelated techniques from Category B.

    c. For cannabis, macroscopic and microscopic examinations will be considered as uncorrelated techniques from Category B when observations include documented details of botanical features. Along with presence of leaves, stems, seeds, and hairs there must be the presence of cystolithic hairs at a minimum to meet acceptance criteria that the plant material is cannabis.

        1) For exhibits of cannabis that lack sufficient observable macroscopic and microscopic botanical detail (e.g. extracts or residues), 9-tetrahydrocannabinol (THC) or other cannabinoids shall be identified utilizing the principles set forth in sections 8.4.2.2.1 and 8.4.2.2.2.

12.4 Reporting the Results

The analyst and technical reviewer confirm that the above criteria are met and agree on the final reported result(s). If there is a discrepancy, the analyst and technical reviewer notify the Forensic Lab Manager. The Forensic Lab Manager will either order additional testing or a qualified third-party is selected to perform a technical review of the results. Third-party reviewers meet the requirements detailed in the Quality Manual, Section 2.2.3. After a third-party review, consensus among the analyst and two reviewers are met in order for a positive result to be reported. If a consensus is not met, then the final results are reported as "Inconclusive" and the Forensic Lab Manager notified. The Forensic Lab Manager in conjunction with the Quality Manager investigates the reason that a result could not be agreed upon and determine whether Corrective Action is initiated.

UNCONTROLLED COPY

12.5 Administrative and Technical Review of the Report

Administrative review of a report is performed by someone trained and authorized to perform the review other than the analyst who prepared the report. The administrative reviewer will:

1) Confirm the technical review of the results is complete.
2) Check that all required information is on the test report and that the case information matches that in the Sample Information Log for each sample or item
3) Review all administrative and examination records to ensure that each page in the records contain unique identifiers for the case.
4) Initial the "AR by" line and date below it

12.6 Finalizing the Report

Once the technical and administrative review is complete, the final report is signed by the analyst.

12.7 Release and Distribution of the Report

The final report is released according to Quality System Manual, QD001, Section 2.10. The report is distributed according to the List of Recipients and Distribution Guidelines, QD010.

UNCONTROLLED COPY

13.0 Maintenance

Refer to the Maintenance Plan SOP, QP102.5 for the schedule of preventive and routine maintenance.

UNCONTROLLED COPY

14.0 References

14.1 Statutes

14.1.1 North Carolina General Statutes Chapter 90, Article 5
14.1.2 North Carolina General Statutes 7A-304(a)(8)

14.2 Laboratory Manuals

14.2.1 Virginia Division of Forensic Sciences, Controlled Substances Procedures Manual
14.2.2 Orange County Crime Laboratory, Controlled Substances Section Manual
14.2.3 North Carolina State Bureau of Investigation, Drug Chemistry Procedure Manual
14.2.4 Pitt County Sheriff Department Crime Laboratory

14.3 Drug Standard References

14.3.1 DEA Drug Logo Index, v 3.31.
14.3.2 O'Neil, Maryadele J., editor. *The Merck Index, Fourteenth Edition.* Whitehouse Station, N.J.: Merck & Co., Inc., 2006.
14.3.3 Marnell, Tim, editor. *Drug Identification Bible, 2010 Edition.*
14.3.4 Mills, Terry, III and Roberson, J. Conrad. *Instrumental Data for Drug Analysis, Third Edition.* New York: CRC Press, 2006.
14.3.5 Moffat, Anthony C et al., editors. *Clarke's Analysis of Drugs and Poisons.* London: The Pharmaceutical Press, 2004.
14.3.6 Rx-ID, electronic version. Amera-Chem Inc., Version 2008.1.

UNCONTROLLED COPY

15.0 Appendices

15.1 Color Test Reference Tables

Commonly Used Color Tests

Color Test	Compound	Result
Duquenois-Levine (Modified)	Marijuana and Hash Oil	Duquenois portion – blue/purple Levine portion – bottom layer pink/purple
Marquis	Opiates (heroin, codeine, morphine)	Purple/violet
	Amphetamine/methamphetamine/ phentermine	Orange to brown
	Aspirin	Pink to deep red on standing
	Phenoxymethylpenicillin	Red
	MDA/MDMA	Black-violet
Cobalt Thiocyanate	Cocaine HCl	Blue precipitate forms
	Cocaine base	Faintly blue – intensifies w/ HCl
	PCP	Blue
	Amitriptyline/doxepin	Blue
	Barbiturates w/ unsaturated side chain (i.e. butalbital)	Faint blue
Secondary amine #2	Secondary amines/ methamphetamine	Blue to violet
	Primary amines/ amphetamine	Slow pink to cherry red

Other Color Tests

Color Test	Compound	Result
Cobalt Thiocyanate (Modified)	Cocaine HCl	Blue in lower $CHCl_3$ layer
Froehde's	Heroin	Purple to green
	Codeine	Green to red/brown
	Morphine	Deep purple to slate
	Aspirin	Blue to purple
	Phenoxymethylpenicillin	Blue
	Pentazocine	Blue
	Acetaminophen	Pale blue
Bates Test	Cocaine base	Very blue precipitate
Stannous Chloride	Cocaine salts	Blue remains
	Cocaine base	Blue color forms
	Other positive compounds	Blue fades
Ferric Chloride	Acetaminophen	Blue
	Salicylamide	Dark purple

UNCONTROLLED COPY

	Hydrolyzed aspirin	Purple
	GHB	Red/brown
Koppanyi	Barbiturates	Red/violet in 30 seconds
Dille-Koppanyi	Barbiturates	Blue purple
	Theophylline, glutethimide	Purple
	Ampicillin	brown
Zwikker	Barbiturates	Purple in organic layer
Meckes	Heroin	Green/blue
	Codeine	Bright-green/blue green
	PCP	Light yellow
	Quinine	Light yellow
Silver Nitrate	Chloride ions	White, curdy precipitate
Barium Chloride	Sulfate containing compounds	Formation of a precipitate
Methanolic KOH	Cocaine	Wintergreen odor
Secondary Amine #1	Secondary amines/ methamphetamine	Yellow/brown in organic phase
Fiegel's/Nitroprusside	Secondary amines	Deep blue color
PDMAB	Primary aromatic amines	Yellow-orange
	Indoles	Purple in less than 30 seconds
Ehrlich's Reagent	LSD, psilocin	Purple
	Benzocaine, procaine	yellow
Benedict's Solution	Ascorbic acid, strong reducing agents, glucose, tetracycline	Red
	Streptomycin	Orange/brown
Chen's Test	Ephedrine, PPA, pseudoephedrine	Purple
Fehlings Solution	Reducing sugars	Yellow to red
GHB Color Test #3 (Smith Test)	GHB	Immediate green color
	Negative results	Pinkish orange
Mayer's Reagent	Alkaloids	White to yellow precipitate
Methylene blue	Vitamin C	Dark blue to light blue
Nitric Acid	Heroin	Yellow green
	Morphine	Red
	Codeine	Orange
	Mescaline	red
	Acetaminophen	Fumes, orange brown
Parri	Barbiturates	Blue
Sulfuric Acid	Tetracycline	Purple to yellow w/ H_2O
	2,3-MDMA, 2,3-MDA	Rose
	3,4-MDMA, 3,4-MDA	Gray-brown
Tannic Acid	Caffeine and theophylline	Precipitate
TBPEE Solutions	Primary amines	Violet

UNCONTROLLED COPY

	Secondary amines	Blue
	Tertiary amine	Red
Van Urk's	LSD	Blue/purple
Weber Test	Psilocyn	Initial red, blue with acid

UNCONTROLLED COPY

15.2 Infrared Spectra Reference Table

Drug Name	Minimum Acceptance Peaks
(+)-Methamphetamine hydrochloride	1450-1490 (2), 690-755 (2), 1050-1090 (2), 1590-1610
(+)-Pseudoephedrine Hydrochloride	690-710, 1020-1040, 1445-1455, 750-770, 1350-1380, 990-1010, 1200-1210, 1580-1600
(±)-3,4-Methylenedioxyamphetamine (MDA)	1480-1510, 1250-1260, 1030-1040, 1430-1450, 790-810, 1180-1200
(±)-3,4-Methylenedioxymethamphetamine Hydrochloride (MDMA)	1480-1510, 1240-1260, 1025-1040, 790-810, 1180-1200, 1435-1450
(±)-Ketamine Hydrochloride	1710-1725, 765-775, 1140-1160, 1035-1045, 1115-1125, 715-725, 645-655
(±)-Methadone hydrochloride	695-715, 1690-1710, 1440-1460, 760-780, 1095-1115, 930-950, 1120-1140
3,4-methylenedioxy-N-ethylamphetamine (MDEA) HCl	1230-1250, 1480-1510, 1030-1050, 1435-1455, 790-810
4-Bromo-2,5-dimethoxyphenethylamine HCl	1190-1210, 1020-1040, 1480-1500, 765-785, 1380-1400
6-monoacetyl morphine	1230-1250, 1720-1740, 1440-1460, 1040-1060, 1020-1040
Allobarbital	1680-1700, 1310-1320, 915-940
Alprazolam	1480-1500, 1600-1620, 690-700, 1306-1326, 1530-1550, 817-837
Amobarbital	1683-1703, 1419-1439, 1302-1418, 1229-1249, 800-820
Barbituric Acid	459-479, 490-510, 763-799, 1238-1360, 1672-1692, 1707-1727
Benzylpiperazine HCl	733-753, 690-710, 1420-1450, 947-967, 1030-1050, 590-610
Bufotenine Oxalate Hydrate	1220-1240, 810-830, 1480-1500, 1600-1630, 780-800, 1040-1060
Butabarbital	1660-1700, 1750-1770, 1290-1320, 1360-1380, 1215-1240, 840-860
Cannabidiol	1210-1220, 1430-1460, 1570-1590, 1620-1640, 1010-1030, 875-900
Cannabinol	1040-1060, 1610-1630, 1390-1410, 1570-1590, 1110-1140, 1210-1240
Cathine HCl	690-710, 750-770, 1030-1050, 1180-1210, 1440-1460, 960-980
Cathinone HCl	680-710, 1680-1700, 1230-1250, 960-990, 1440-1460, 1590-1610

UNCONTROLLED COPY

Cocaine hydrochloride	1700-1720, 1720-1740, 1260-1280, 1100-1120, 720-740, 1060-1080, 1220-1240
Cocaine base	700-720, 1100-1120, 1020-1050, 1260-1280, 1720-1740, 1690-1720
Codeine	1040-1060, 1260-1290, 1490-1510, 1100-1130, 780-810, 920-950
D-Amphetamine hemisulfate salt	1040-1060, 610-620, 590-610, 690-710, 1020-1030, 730-750, 1530-1550, 1380-1400
delta-9-Tetrahydracannabinol (THC)	1030-1050, 1075-1095, 870-890, 680-700, 1415-1435, 1590-1620
Diazepam	1675-1695, 695-715, 1300-1325, 730-750, 830-850, 1120-1140, 1470-1495, 1330-1350
Ephedrine Hydrochloride	690-710, 745-765, 980-1000, 1050-1070, 670-690, 1345-1410, 1455-1475
Fentanyl HCl	1635-1665, 690-715, 1265-1285, 1240-1260, 1390-1410, 1370-1390
Flunitrazepam	1690-1710, 1610-1630, 1480-1500, 1520-1540, 1100-1120, 770-795
gamma-Butyrolactone (GBL)	1750-1770, 1150-1170, 1020-1040, 860-880, 980-1000, 1365-1385
Gamma-hydroxybutric Acid (GHB) Sodium Salt	1005-1025, 1540-1560, 1395-1415, 1055-1075, 910-930, 1440-1460
Glutethimide	1670-1700, 1190-1210, 1260-1280, 1340-1360, 690-710, 750-770
Heroin base	1180-1200, 1220-1240, 1730-1750, 1025-1045, 1750-1770, 1355-1375, 1440-1460
Hydrocodone Bitartrate Salt	1260-1280, 1120-1140, 660-680, 785-805, 1045-1065, 1490-1510, 1435-1455, 1695-1715
Hydromorphone hydrochloride	730-750, 1300-1320, 1705-1725, 965-985, 1010-1030, 1490-1510, 1260-1280
Levomethorphan	1210-1250, 1000-1050, 1400-1500, 800-900, 1600-1650, 1500-1550
Lorazepam	1675-1710, 1120-1160, 1305-1335, 740-760, 1470-1490, 1590-1615
Lysergic Acid Diethylamide (LSD)	740-760, 1610-1630, 1435-1455, 765-785, 1205-1225, 1040-1080
Meperidine	690-710, 1210-1230, 720-740, 1145-1165, 1090-1110, 970-990
Mephedrone HCl	1675-1695, 820-840, 1240-1260, 1590-1615, 965-985, 725-745
Meprobamate	1680-1700, 1060-1080, 1390-1410, 1330-1350, 1580-1600, 775-795

UNCONTROLLED COPY

Mescaline	1110-1130, 1190-1210, 560-580, 1015-1035, 880-900, 1410-1430
Methandrostenolone	1650-1670, 1610-1630, 875-895, 1590-1610, 1150-1170, 1360-1380
Methylaminopropiophenone HCl (methcathinone)	1680-1700, 690-710, 1235-1255, 1440-1460, 965-985, 1590-1610
Methylenedioxy pyrovalerone HCl	1240-1260, 1020-1040, 1675-1695, 1090-1110, 1425-1445, 920-940
Methylone HCl	1250-1270, 1080-1100, 1440-1460, 1670-1690, 920-940, 870-900
Methylphenidate hydrochloride	690-710, 1730-1750, 1160-1180, 725-745, 1190-1220, 1140-1160, 1420-1440
Morphine sulfate salt pentahydrate	790-810, 1110-1130, 1240-1260, 1440-1490, 940-960, 1070-1090
n,n-Diisopropyl-5-methoxytryptamine	790-810, 1210-1230, 1420-1440, 920-940, 1060-1080, 1480-1500
Nandrolone	1640-1660, 1060-1080, 1120-1140, 870-890, 1190-1210, 1250-1280
Oxazepam	1680-1700, 1700-1725, 1120-1140, 690-710
Oxycodone hydrochloride	1020-1040, 1265-1285, 930-950, 1430-1450, 1490-1515, 1710-1745
Pentazocine HCl	1210-1230, 1260-1280, 1430-1460, 1490-1510, 1600-1620, 920-940
Pentobarbital	1660-1680, 1310-1335, 1280-1300, 840-860, 1365-1385
Phencyclidine hydrochloride	690-710, 750-770, 1435-1455, 885-905, 1005-1025, 1285-1305
Phendimetrazine bitartrate	1110-1140, 1080-1110, 690-710, 1690-1710, 990-1010, 740-770
Phenobarbital sodium salt	1550-1650, 1285-1310, 1260-1280, 1335-1345, 1650-1670, 680-700, 1410-1440
Phentermine HCl	720-740, 690-710, 1270-1290, 1480-1500, 1370-1390, 1600-1620
Procaine	1265-1285, 1680-1700, 1100-1125, 760-780, 1155-1180, 1590-1610
d-Propoxyphene HCl	1710-1730, 690-710, 1170-1190, 955-975, 715-735, 750-780
Psilocin	820-840, 1250-1270, 1220-1240, 1020-1045, 690-750, 1330-1350
Psilocybin	910-940, 1035-1060, 1090-1110, 1220-1240, 1490-1510, 730-755
Secobarbital	1550-1570, 1330-1340, 1260-1280, 1300-1320, 1645-1665, 1430-1450, 1690-1710, 770-790

UNCONTROLLED COPY

Stanozolol	1040-1060, 1075-1095, 920-940, 790-810, 1360-1380, 1435-1455
Talbutal	1670-1710, 1350-1370, 1420-1440, 1200-1220, 830-850, 1740-1760
Testosterone Propionate	1650-1670, 1710-1730, 850-870, 1230-1250, 1010-1030, 1060-1080
Trifluoromethylphenylpiperazine	1110-1130, 1065-1085, 1290-1310, 1580-1600, 1440-1460, 780-800

UNCONTROLLED COPY

15.3 Mass Spectra Reference Table

Drug Name	$[M]^+$ or $[M-H]^+$ (^{13}C Peak)	Major Peaks
3,4-Methylenedioxyamphetamine (MDA)	178(179)	44, 136, 77, 51
3,4-Methylenedioxymethamphetamine (MDMA)	192(193)	158, 135, 77, 136
3,4-methylenedioxy-N-ethylamphetamine (MDEA)	207(208)	72, 44, 135, 77
3-Fluoromethcathinone	181 not present need Rt	58, 95, 75, 123
4-Bromo-2,5-dimethoxyphenethylamine	260(261)	230, 77, 215, 105
6-monoacetyl morphine	327(328)	268, 215, 147, 115
Allobarbital	208(209)	167, 124, 80, 166
Alprazolam	308(309)	279, 273, 204, 77
AM-2201	359(360)	232, 127, 284, 342
Amobarbital	226(227)	156, 141, 41, 55
Benzylpiperazine	181 not present need Rt	91, 134, 176, 56
Bufotenine	204(205)	58, 146, 42, 159
Butabarbital	212(213)	141, 156, 98, 57
Cannabidiol	314(315)	231, 246, 174, 121
Cannabinol	310(311)	295, 238, 223, 165
Cathine	150 not present need Rt	44, 77, 79, 51
Cathinone	148(149)	44, 77, 51, 105
Cocaine hydrochloride	303(304)	82, 182, 77, 105
Codeine	299(300)	162, 42, 115, 229
D-Amphetamine	134(135)	44, 91, 65, 120
delta-9-Tetrahydracannabinol (THC)	314(315)	299, 231, 271, 243
Diazepam	284(285)	256, 283, 221, 255
Ephedrine HCl	165 not present need Rt	58, 77, 51, 79
Fentanyl	336 not present need Rt	245, 146, 42, 189
Flunitrazepam	313(314)	285, 312, 266, 238
gamma-Butyrolactone (GBL)	86(87)	42, 41, 56, 85
Gamma-hydroxybutric Acid (GHB)	104(103)	42, 86, 41, 56
Glutethimide	217(218)	189, 117, 132, 160
Heroin	369(370)	327, 268, 310, 204
HU-210	386(387)	302, 287, 43, 316, 330
Hydrocodone	299(300)	242, 214, 96, 185
Hydromorphone	285(286)	228, 229, 214, 115
JWH-015	327(328)	326, 310, 270, 127
JWH-018	341(342)	284, 214, 324, 340
JWH-019	355(356)	284, 228, 127, 338
JWH-073	327(328)	284, 200, 310, 127
JWH-081	371(372)	354, 314, 214, 185
JWH-122	355(356)	298, 214, 338, 144
JWH-200	384(385)	100, 127, 56, 155

UNCONTROLLED COPY

JWH-203	339(340)	214, 144, 213, 116
JWH-210	369(370)	352, 312, 214, 144
JWH-250	335(336)	214, 144, 215, 116
JWH-398	375(376)	214, 318, 358, 144
Ketamine	237 not present need Rt	180, 182, 209, 152
LAMPA (Lysergic Acid Methyl Propyl Amide)	323(324)	221, 207, 181, 196
Levomethorphan	271(272)	59, 150, 214, 171
Lorazepam	321 not present need Rt	239, 274, 75, 276
Lysergic Acid Diethylamide (LSD)	323(324)	221, 207, 181, 222
Meperidine	247(248)	71, 70, 42, 57, 172
Mephedrone	176(177)	58, 91, 65, 119
Meprobamate	218 not present need Rt	55, 83, 43, 71
Mescaline	211(212)	182, 167, 181, 151
Methadone	309 not present need Rt	72, 165, 223, 115
Methamphetamine	148(149)	58, 91, 56, 134
Methandrostenolone	300(301)	122, 121, 91, 161
Methcathinone	163 not present need Rt	58, 77, 51, 56
Methylenedioxy pyrovalerone	275 not present need Rt	126, 127, 149, 96
Methylone	207(208)	58, 65, 149, 121
Methylphenidate	233 not present need Rt	84, 91, 150, 55
Morphine	285(286)	162, 215, 115, 174
n,n-Diisopropyl-5-methoxytryptamine	274(275)	114, 72, 160, 145
Nandrolone	274(275)	110, 91, 79, 105
Oxazepam	286 not present need Rt	267, 268, 205, 239
Oxycodone	315(316)	230, 201, 258, 115
Pentazocine	285(286)	217, 110, 70, 202
Pentobarbital	226 not present need Rt	141, 156, 157, 55
Phencyclidine	243(244)	200, 91, 242, 186
Phendimetrazine	191(192)	85, 57, 42, 56
Phenobarbital	232(233)	204, 117, 232, 161
Phentermine	149 not present need Rt	58, 91, 65, 134
Propoxyphene	339 not present need Rt	58, 91, 115, 130
Pseudoephedrine	165 not present need Rt	58, 105, 77, 106
Psilocin	204(205)	58, 77, 117, 146
Psilocybin	284(285)	58, 204, 159, 146
Salvinorin A	432(433)	94, 121, 107, 95
Secobarbital	238 not present need Rt	168, 167, 153, 97
Stanozolol	328(329)	96, 94, 257, 270
Talbutal	224 not present need Rt	167, 168, 97, 124
Testosterone Propionate	344(345)	57, 124, 147, 91
Trifluoromethylphenylpiperazine	230(231)	188, 56, 145, 172

UNCONTROLLED COPY

16.0 Revision Table

Revision #	Effective date	Revised by	Description of Revisions
Original	05/04/2011	B. Pridgen	
#1	11/19/2012	A. Hutson	Addition of Training Module
		B. Pridgen	Reformatted entire document. Rearranged sections to comply with QSM & QP101.13. Removed repetitive text that is in QSM. Added more detail to tech/admin review. Changed verb tense throughout.
#2	03/27/2013	A. Hutson B. Pridgen	Broad revisions of entire SOP to include addition of more specific acceptance criteria for QC checks and testing methods, reference tables in appendix, grammatical changes for consistency throughout SOP, training completion requirements and a re-training module.
#3	04/24/2013	B. Pridgen	Fixed typos in Table 10.6. RF Frequency <2000 kHz and Prefilter Offset >-11 V.
#4	11/6/2015	A. Hutson B. Pridgen	Addition of court testimony training and uncertainty of measurement requirements. Update to acceptance criteria for suspected marijuana samples.

UNCONTROLLED COPY

Authorization

This Standard Operating Procedure, Revision Issue #4, has been approved and authorized by:

Bethany P. Pridgen, MFS
Forensic Lab Director

Date

Ralph M. Evangelous
Chief of Police

Date

UNCONTROLLED COPY