



---

# ARKANSAS STATE CRIME LABORATORY FORENSIC CHEMISTRY QUALITY MANUAL

---

# CONTENTS

1	SCOPE .....	6
1.1	International Standard: General Requirements .....	6
1.2	International Standard: Scope .....	6
1.2.1	ANAB Program .....	6
2	NORMATIVE REFERENCES .....	7
3	TERMS AND DEFINITIONS .....	8
4	GENERAL REQUIREMENTS .....	9
4.1	Impartiality .....	9
4.2	Confidentiality .....	9
5	STRUCTURAL REQUIREMENTS .....	10
5.1	Establishment .....	10
5.2	Management .....	10
5.2.6	Other Staff (Forensic Chemistry Staff) .....	10
5.3	Scope Of Laboratory Activities .....	14
5.4	Normative Documents .....	14
5.5	Laboratory Operations .....	14
5.6	Quality Management .....	14
5.7	Management System Communication And Integrity .....	14
6	RESOURCES REQUIREMENTS .....	15
6.1	General .....	15
6.2	Personnel .....	15
6.2.1	General .....	15
6.2.2	Competence Requirements .....	15
6.2.3	Competence Of Staff .....	15
6.2.4	Duties, Responsibilities, And Authorities .....	15
6.2.5	Personnel Requirements .....	16
6.2.6	Authorizations .....	16
6.3	Facilities And Environmental Conditions .....	16
6.3.1	General .....	16
6.3.2	Documentation .....	16
6.3.3	Monitoring Records .....	16
6.3.4	Control Of Facilities .....	16
6.3.5	External Activities .....	18
6.4	Equipment .....	18
6.4.1	Access .....	18
6.4.2	Outside Equipment .....	18
6.4.3	Proper Functioning .....	19
6.4.4	Performance Verification .....	23
6.4.5	Fitness For Service .....	23
6.4.6	Calibration Requirement .....	23
6.4.7	Calibration Program .....	24
6.4.8	Labelling .....	25
6.4.9	Out Of Service .....	25
6.4.10	Intermediate Checks .....	25
6.4.11	Correction Factors .....	25
6.4.12	Equipment Adjustment .....	25
6.4.13	Equipment Records .....	25

6.5	Metrological Traceability.....	25
6.6	Externally-Provided Products And Services .....	25
7	PROCESS REQUIREMENTS.....	27
7.1	Review Of Requests, Tenders, And Contracts.....	27
7.1.1	General.....	27
7.1.2	Inappropriate Requests.....	27
7.2	Selection, Verification, And Validation Of Methods.....	27
7.2.1	Selection And Verification Of Methods .....	27
7.2.2	Validation Of Methods.....	27
7.3	Sampling .....	27
7.3.1	General.....	28
7.3.2	Sample Selection/Collection.....	30
7.4	Handling Of Test Items .....	33
7.4.1	Evidence Storage .....	33
7.4.2	Item Identification .....	35
7.4.3	Deviations.....	35
7.5	Technical Records.....	36
7.5.1	Case Notes .....	36
7.5.2	Amendments To Technical Records.....	39
7.6	Evaluation Of Measurement Uncertainty .....	40
7.7	Ensuring The Validity Of Results.....	40
7.7.2	Interlaboratory Comparisons .....	41
7.7.3	Monitoring Activity Analysis .....	41
7.7.4	Individual Performance Monitoring .....	41
7.7.5	Performance Monitoring Requirements .....	41
7.7.6	Performance Monitoring Schedule.....	42
7.7.7	Proficiency Test Sourcing.....	42
7.7.8	Performance Monitoring Records.....	42
7.7.9	Re-examination Policy .....	42
7.8	LANGUAGE FOR REPORTS AND TESTIMONY .....	43
7.8.1	General.....	43
7.8.2	Common Requirements For Reports.....	44
7.8.3	Specific Requirement For Test Reports.....	49
7.8.4	Specific Requirements For Calibration Certificates .....	50
7.8.5	Reporting Sampling – Specific Requirements.....	50
7.8.6	Reporting Statements of Conformity.....	50
7.8.7	Reporting Opinions and Interpretations.....	50
7.8.8	Amendments to Reports .....	50
7.8.9	Supplemental Reports.....	51
7.8.10	retesting reports.....	51
7.8.11	Language for Testimony.....	51
7.9	Complaints .....	53
7.10	Nonconforming Work .....	53
7.11	Control Of Data And Information Management.....	53
8	MANAGEMENT SYSTEM REQUIREMENTS.....	54
9	TEST METHODS.....	55
9.1	Testing Requirements.....	55
9.1.1	Minimum Testing Per Exhibit.....	55
9.2	Weight Measurement.....	59
9.2.1	Scope.....	59

9.2.2	Reagents/Standards/Controls .....	59
9.2.3	Sample Preparation.....	59
9.2.4	Quality Assurance/Control Measures .....	62
9.2.5	Interpretation Of Results .....	63
9.2.6	Documentation Requirements .....	63
9.3	Gas Chromatography-Mass Spectrometry (GCMS) .....	64
9.3.1	Scope.....	64
9.3.2	Reagents, Standards And Controls.....	64
9.3.3	Sample Preparation And Acquisition .....	64
9.3.4	Quality Assurance/Control Measures .....	65
9.3.5	Interpretation Of Results And Required Documentation .....	68
9.3.6	Required Documentation For GCMS Results.....	72
9.4	Gas Chromatography-Retention Time (GCRT).....	73
9.4.1	Scope.....	73
9.4.2	Reagents, Standards, And Controls.....	73
9.4.3	Sample Preparation And Acquisition .....	74
9.4.4	Quality Assurance/Control Measures .....	74
9.4.5	Interpretation Of Results .....	74
9.4.6	Required Documentation For GC Qualitative Results .....	75
9.5	Fourier Transform Infrared Spectroscopy (FTIR).....	76
9.5.1	Scope.....	76
9.5.2	Reagents, Standards, And Controls.....	76
9.5.3	Sample Preparation And Acquisition .....	77
9.5.4	Quality Assurance/Control Measures .....	78
9.5.5	Interpretation Of Results .....	79
9.5.6	Documentation Requirements .....	80
9.6	Thin Layer Chromatography.....	81
9.6.1	Scope.....	81
9.6.2	Reagents, Standards, And Controls.....	81
9.6.3	Sample Preparation And Acquisition .....	82
9.6.4	interpretation of the results .....	84
9.6.5	Documentation Requirements .....	85
9.7	Pharmaceutical Identification .....	86
9.7.1	Scope.....	86
9.7.2	Reagents, Standards, And Controls.....	86
9.7.3	Sample Preparation.....	86
9.7.4	Quality Assurance/Control Measures .....	87
9.7.5	Interpretation Of Results .....	87
9.7.6	Documentation Requirements .....	87
9.8	Color Testing.....	88
9.8.1	Scope.....	88
9.8.2	Reagents, Standards, And Controls.....	89
9.8.3	Sample Preparation And Acquisition .....	89
9.8.4	Quality Assurance/Control Measures .....	91
9.8.5	Interpretation Of Results .....	91
9.8.6	Documentation Requirements .....	92
9.9	Morphological Microscopy Of Plant Material .....	92
9.9.1	Scope.....	92
9.9.2	Reagents, Standards, And Controls.....	92
9.9.3	Sample Preparation.....	92

9.9.4	Quality Assurance/Control .....	92
9.9.5	Interpretation Of The Results .....	92
9.10	Semi-Quantitative Determination of $\Delta^9$ -THC .....	93
9.10.1	Scope.....	93
9.10.2	Reagents, Standards, and Controls .....	93
9.10.3	Quality Assurance/Control Measures .....	95
9.10.4	Interpretation of Results.....	96
9.10.5	Required Documentation.....	97
9.11	Extractions For Difficult Samples.....	97
9.11.1	Required Extractions.....	97
9.11.2	Suggested Extractions For Difficult Samples.....	98

# 1 SCOPE

This manual follows the requirements specified by ANSI-ASQ National Accreditation Board (ANAB), which is based on the ISO/IEC 17025:2017 standards and the 2017 ANAB ISO/IEC 17025:2017 — Accreditation Requirements for Forensic Testing and Calibration (2023) (AR 3125).

The *Forensic Chemistry Quality Manual* is written specifically for the analysts working in the Drug Section and performing analyses in the following areas:

- Controlled Substance Analysis
- Clandestine Laboratory Analysis
- General Chemical Testing
- Tampering

Evidence selection, for analysis, is based on the analyst's training and experience.

## 1.1 INTERNATIONAL STANDARD: GENERAL REQUIREMENTS

See *ASCL-DOC-01 Quality Manual*.

## 1.2 INTERNATIONAL STANDARD: SCOPE

See *ASCL-DOC-01 Quality Manual*.

### 1.2.1 ANAB PROGRAM

---

See *ASCL-DOC-01 Quality Manual*.

## 2 NORMATIVE REFERENCES

The Forensic Chemistry section follows applicable references listed in *ASCL-DOC-01 Quality Manual*.

Additional references include:

- *ASCL-DOC-01 Quality Manual*
- Arkansas Criminal Code for Controlled Substances
- Arkansas Controlled Substance List

### 3 TERMS AND DEFINITIONS

Additions to *ASCL-DOC-01 Quality Manual* are listed below.

#### CONTROLLED SUBSTANCE

A drug or chemical, whose manufacture, possession, or use is regulated by a government.

#### CUTTING AGENT (DILUENT, ADULTERANT)

A substance added to reduce the purity of another substance.

#### EVIDENCE

Items submitted to the laboratory for analysis.

## 4 GENERAL REQUIREMENTS

### 4.1 IMPARTALITY

See *ASCL-DOC-01 Quality Manual*.

### 4.2 CONFIDENTIALITY

Investigative information may not be released until after a technical review has been completed. Should information need to be released, before the case is completed and a technical review has been conducted, the following shall occur:

- Separate competent individual shall review data to confirm findings to be released (e.g., GCMS, FTIR, TLC, GCRT results)
- Reviewer of data shall document what was reviewed (this may be done by memo or via an email to the case analyst stating what data was reviewed and what may be released)
- During the release of information, the analyst shall communicate their results are preliminary and a final report shall be completed and released upon completion of analysis

## 5 STRUCTURAL REQUIREMENTS

### 5.1 ESTABLISHMENT

Act 517 of 1977 established the Arkansas State Crime Laboratory (ASCL) via A. C. A. § 12-12-301.

### 5.2 MANAGEMENT

The Arkansas State Crime Laboratory is managed by the Director, who has overall responsibility for the laboratory.

For 5.2.1 – 5.2.5 See *ASCL-DOC-01 Quality Manual*.

#### 5.2.6 OTHER STAFF (FORENSIC CHEMISTRY STAFF)

---

##### 5.2.6.1 CHIEF FORENSIC CHEMIST

###### QUALIFICATIONS

The position requires a baccalaureate or advanced degree in natural science or a closely related field with knowledge of the principles and practices of chemistry, chemical analysis, and laboratory equipment; plus five years of experience in a chemical laboratory, including two years in a forensic laboratory, or a related field. Other job-related education and/or experience may be substituted for all or part of these basic requirements upon approval of the Deputy Director.

###### AUTHORITIES AND RESPONSIBILITIES

The Chief Forensic Chemist is under administrative direction and is responsible for the activities of the Forensic Drug Chemistry Section in Little Rock and satellite laboratories. The Chief Forensic Chemist has the overall responsibility for the technical operations and the provision of the resources needed to ensure the quality of the laboratory operations. The Chief Forensic Chemist will have the appropriate technical training and technical experience in the drug section. The Chief Forensic Chemist will have regular contact with crime laboratory staff, frequent contact with law enforcement agencies and judicial officials, and limited contact with the public. The Chief Forensic Chemist ensures compliance with ANAB International requirements by implementing lab wide policies and overseeing the section's quality assurance program.

- Supervises a large-sized technical staff of Forensic Chemists including interviewing applicants and recommending for hire, approving leave, making work assignments, training employees and evaluating the performance of employees
- Assists with developing laboratory policies and procedures, develops short and long-range operational plans for the forensic chemistry section, monitors operational activities by conducting staff meetings to disseminate information and reviewing and approving reports and compiles and submits statistical reports

- Manages the controlled substances authorized by the Drug Enforcement Administration (DEA) to be used during the process for comparing pure samples of controlled substances with findings to establish standards and maintains a log of the controlled substances used during testing including dates, amounts, and the name of the chemist requisitioning the substance
- Performs qualitative and quantitative forensic chemical analysis of known and unknown substances received from law enforcement agencies to determine the content of the substances using standardized laboratory methods and instruments, and documents procedure and results
- Presents expert forensic testimony in court on the chemical analytical methodology used to analyze evidence and analysis results, supervises pretrial conferences, and provides consultation to law enforcement and judicial officials on evidence collection and preservation method
- Compiles and interprets data obtained from analytical instruments, reviews and approves scientific forensic reports of section chemists, and writes conclusive scientific forensic reports
- Provides classroom instruction for law enforcement officers at seminars and courses statewide on drug identification, collection of evidence, and clandestine drug laboratory investigations
- Conducts research studies and validates new forensic analytical procedures, reviews current scientific literature and attends and participates in meetings and seminars to keep abreast of new technologies and procedures in the field.
- Performs related responsibilities as required or assigned

#### **5.2.6.2 TECHNICAL LEADER**

---

The Technical Leader is in charge of and accountable for the quality and training for the Forensic Chemistry section. The Technical Leader serves in this role for both the Little Rock and Lowell locations and is assisted by the training officers at each location.

#### **QUALIFICATIONS**

The position requires a baccalaureate or advanced degree in natural science or a closely related field with knowledge of the principles and practices of chemistry, chemical analysis, and laboratory equipment; plus three years of experience in a chemical laboratory, including two years in a forensic laboratory, or a related field. Other job-related education and/or experience may be substituted for all or part of these basic requirements upon approval of the Deputy Director. The technical lead is in charge of quality and training for the Forensic Chemistry Section.

#### **NEW TECHNICAL LEADER APPOINTMENTS**

Any Forensic Chemistry Technical Leader appointed on or after September 1, 2022 shall be a currently or previously qualified analyst in each technology utilized in the section, or have documented training in each technology utilized in the section within one year of appointment.

Newly appointed technical leaders shall be responsible for the following within one year of appointment:

- 1) Review of validation studies and analytical procedures currently used by the section
- 2) Review of educational and training records of currently qualified analysts and technical reviewers
- 3) ANAB assessor training (could be longer than 1 year depending on availability of training)

## AUTHORITIES AND RESPONSIBILITIES

- All duties of Forensic Chemist
- Ensures that instrument, balance, chemical/reagent and reference material logs are recorded appropriately; prepares and records proficiency tests/intralaboratory comparisons; maintains reference material inventory
- Helps maintain and update the section's manuals and documents
- Monitors the section's practices for compliance with the section's SOP
- Ensures the validation and verification of new technical procedures
- Works with lab wide Quality Manager and Chief Forensic Chemist to seek ways to improve the quality system
- Oversees the training program and training officers
- Works with Chief Forensic Chemist to seek ways to improve the training program.
- Coordinates intern program projects

## AUTHORIZATIONS

- Can recommend suspension and resumption of Forensic Chemistry technical operations for the laboratory or an individual
- Oversees the technical operations of the Forensic Chemistry section
- Approves method development, modification, verification, and/or validation

### 5.2.6.3 FORENSIC CHEMIST

---

#### QUALIFICATIONS

The Forensic Chemist must possess a baccalaureate or advanced degree in natural science or closely related field with knowledge of the principles and practices of chemistry, chemical analysis and laboratory equipment. Before performing casework, the forensic chemist will be required to successfully complete a training program that will include competency sample testing, written and oral examination, and a mock trial (this training program is waived for forensic chemists hired before the issuance of the quality program and can be modified, based on experience, for those hired after the issuance of the quality program). This position is governed by state and federal laws and agency policy.

#### AUTHORITIES AND RESPONSIBILITIES

- Process evidence suspected of containing controlled substance(s) submitted to the ASCL by law enforcement agencies

- Present expert forensic testimony in court on chemical analytical methodology used to analyze evidence and obtain results
- Participate in pretrial conferences and provide consultation to law enforcement and judicial officials on evidence collection, preservation methods and analysis results.
- Verify the correct operation of scientific instruments and perform routine maintenance as needed. Prepare and verify reference materials and reagents according to established guidelines
- Review current scientific literature. Study and validate new forensic analytical procedures and modify new and/or old procedures as necessary
- Attend and participate in professional meetings and seminars to keep abreast of new technologies and methods in chemistry
- Assist with training new laboratory staff in performing standardized laboratory test.
- Perform related responsibilities as required or assigned
- Process evidence suspected of containing controlled substance(s) or chemicals that are suspected of being used to manufacture a controlled substance submitted to the ASCL by law enforcement agencies (Illicit Lab chemist only)
- On call to aid law enforcement in safely dismantling illicit laboratories and collecting representative samples (Illicit Lab chemist only)
- Instruct law enforcement officials on proper methods of confiscating, preserving, and disposing of toxic chemicals, waste and equipment found in clandestine drug laboratories (Illicit Lab chemist only)

#### **5.2.6.4 FORENSIC TECHNICIAN**

---

##### **QUALIFICATIONS**

This position requires the formal education equivalent of a high school degree.

##### **AUTHORITIES AND RESPONSIBILITIES**

- Perform performance verifications on instrumentation
- Transport evidence between Secure Storage and FC Secure Storage
- Assess submission sheets
- Prepare chemicals, reagents, reference materials, and controls needed for casework
- Assign daily reviews to Forensic Chemistry personnel
- Conduct annual inventory of controlled reference materials
- Perform related responsibilities as required or assigned
- Sampling of bulk cases, when trained and authorized

#### **5.2.6.5 HEALTH AND SAFETY OFFICER**

- Conducts monthly safety inspections and ensuring that proper practices and procedures are being followed in the section

- Assists with safety duties assigned by lab wide Health and Safety Manager
- Works with the lab wide Health and Safety Manager to seek ways to improve the safety program

#### 5.2.6.6 TRAINING OFFICER

---

- Makes sure reading material for new hires is relevant and up to date
- Helps maintain and update the training manual and forms
- Devises a training schedule for new hires
- Works with Technical Leader to generate ideas for continued training for the section and ways to improve the training program for new hires
- Makes competency samples for new hires

### 5.3 SCOPE OF LABORATORY ACTIVITIES

See *ASCL-DOC-01 Quality Manual*.

### 5.4 NORMATIVE DOCUMENTS

See *ASCL-DOC-01 Quality Manual*.

### 5.5 LABORATORY OPERATIONS

See *ASCL-DOC-01 Quality Manual*.

### 5.6 QUALITY MANAGEMENT

See *ASCL-DOC-01 Quality Manual*.

### 5.7 MANAGEMENT SYSTEM COMMUNICATION AND INTEGRITY

See *ASCL-DOC-01 Quality Manual*.

## 6 RESOURCES REQUIREMENTS

### 6.1 GENERAL

See *ASCL-DOC-01 Quality Manual*.

### 6.2 PERSONNEL

#### 6.2.1 GENERAL

---

See *ASCL-DOC-01 Quality Manual*.

#### 6.2.2 COMPETENCE REQUIREMENTS

---

See *ASCL-DOC-01 Quality Manual*.

##### 6.2.2.1 ANALYST/EXAMINER EDUCATIONAL REQUIREMENTS

---

See *ASCL-DOC-01 Quality Manual*.

##### 6.2.2.2 TRAINING PROGRAM

---

The Forensic Chemistry Training Program is normally completed over a minimum of thirty-nine weeks. For analysts with prior experience, the training may be truncated with the approval of the Section Chief and the Deputy Director.

See *ASCL-DOC-01 Quality Manual*, *DRG-DOC-02 Training Manual*, and *ASCL-DOC-03 ASCL New Analyst/Technician Training Manual*.

The Forensic Chemistry Section encourages the distribution and review of current literature. To this end, a literature folder is provided on the shared Drug network drive, to which literature is periodically added. Additionally, new literature may be distributed by email.

#### 6.2.3 COMPETENCE OF STAFF

---

See *ASCL-DOC-01 Quality Manual* and *DRG-DOC-02 Training Manual*.

##### 6.2.3.1 COMPETENCY TESTING

---

See *ASCL-DOC-01 Quality Manual* and *DRG-DOC-02 Training Manual*.

##### 6.2.3.2 COMPETENCY-TESTED ACTIVITIES

---

See *ASCL-DOC-01 Quality Manual* and *DRG-DOC-02 Training Manual*.

## 6.2.4 DUTIES, RESPONSIBILITIES, AND AUTHORITIES

---

See job descriptions in section 5.2 of this manual.

## 6.2.5 PERSONNEL REQUIREMENTS

---

See *ASCL-DOC-01 Quality Manual*.

## 6.2.6 AUTHORIZATIONS

---

See *ASCL-DOC-01 Quality Manual*.

## 6.3 FACILITIES AND ENVIRONMENTAL CONDITIONS

### 6.3.1 GENERAL

---

See *ASCL-DOC-01 Quality Manual*.

### 6.3.2 DOCUMENTATION

---

See *ASCL-DOC-01 Quality Manual*.

### 6.3.3 MONITORING RECORDS

---

A record of the temperature conditions for all Certified Reference Material storage locations within the section will be maintained (*DRG-FORM-47 Temperature Log*). Storage location conditions should be below 0°C. If storage conditions deviate from that specification for an extended time period, the cause will be assessed and any necessary action will be taken. Thermometers are assessed and replaced as needed.

### 6.3.4 CONTROL OF FACILITIES

---

See *ASCL-DOC-01 Quality Manual*.

#### 6.3.4.1 ACCESS

---

#### LITTLE ROCK

The Forensic Chemistry section has a Secure Storage room within the section for storage of evidence prior to case assignment. This area is accessible by fob; those fobs are assigned to Forensic Chemists, Forensic Chemistry Section Chiefs/Technical Lead, and the Forensic Chemistry Technician.

Each Forensic Chemist has lockable areas to store assigned casework evidence. The Chief Forensic Chemist has access to these storage areas.

The Forensic Chemistry section has a key box containing cabinet keys and sections door keys. The key to the section key box is kept by the Chief Forensic Chemist. A log must be kept when keys are added or removed from the section key box.

Reference materials (i.e., drug standards) are kept in a locked filing cabinet. Keys for the filing cabinet are assigned to Chief Forensic Chemist and Chief Forensic Toxicologist. The Chief Forensic Chemist may designate other personnel as needed. A logbook of all transactions will be kept and an inventory of all controlled reference materials will be conducted as needed. The return of reference materials to the storage location shall be witnessed by another individual and documented on *DRG-FORM-42 Chronological Log Sheet for Drug Reference Material*.

## LOWELL REGIONAL LABORATORY

Each Forensic Chemist has lockable areas to store assigned casework evidence. The back doors at each facility are for maintenance and emergency uses only. These doors shall not be used for daily entrance and exit of the building.

There is a key box containing cabinet keys and section's door keys. The key to the section key box is kept by the Chief Forensic Chemist. A log must be kept when keys are added or removed from the section key box.

Reference materials (i.e., drug standards) are kept in a locked cabinet. A key to the locked cabinet is assigned to the Chief Forensic Chemist. The Chief Forensic Chemist can assign a key to other personnel as needed. A logbook of all transactions will be kept and an inventory of all controlled reference materials will be conducted as needed. The return of reference materials to the storage location shall be witnessed by another individual and documented on *DRG-FORM-42 Chronological Log Sheet for Drug Reference Material*.

### 6.3.4.2 PREVENTION OF ADVERSE INFLUENCES

The Forensic Chemistry section has multiple measures in place to prevent contamination, cross-contamination, and adverse influence on laboratory activities.

Analysts shall make every effort to maintain a clean, contamination free, workspace. Materials and consumables used in the sampling process and/or analysis shall be stored in closed containers, cabinets, or drawers. These materials include but are not limited to:

- weigh papers/vessels
- pipettes
- test tubes
- vials
- beakers

- capillary tubes
- autosampler vials and caps

Floors and work surfaces should be kept as free of clutter as possible. All surfaces and equipment within the laboratory, where processing and/or analysis occur, should be regarded as potentially contaminated and should be cleaned on a daily basis.

Balances will be checked daily, prior to use, for cleanliness. The weigh pan should be removed and the balance cleaned prior to performance verification for the day. Throughout the day, any visible residue must be removed prior to weighing case samples. Exterior surfaces of equipment to include balances, vortex mixers, microscopes, and heat sealers, should be wiped as appropriate.

Analysts shall ensure that the work area is clean prior to opening an evidence item and after processing each evidence item. Items selected for analysis will be sampled one at a time and sampling materials to include scissors, tweezers, spatulas, and box cutters, will be cleaned with an appropriate solvent between each sampling. Items selected for analysis should be sampled and immediately sealed or maintained in the analysts' short term storage lockers under a temporary seal. A temporary seal shall be such that the contents cannot readily escape. If any evidence is spilled or dropped, the analyst should check the evidence to ensure that all material/units are present. The analyst shall record the occurrence in the case file and clean the area thoroughly to ensure that contamination of other evidence and the work area does not occur.

General clean-up of work areas should be performed at the close of each work day. In addition to policies listed above, the following schedule should be followed:

- Daily cleaning: bench, equipment, utensils
- Bi-weekly cleaning: test tube racks, vial trays
- Monthly cleaning: lab coats, inner surfaces (drawers, cabinets, areas where consumables are stored), outer surfaces (drawers, cabinets, sink, window sills and chairs), wipe all surfaces, including partitions, windows, doors and handles
- Bi-annual cleaning: inner storage areas (remove the entire contents of cabinets and drawers), fume hoods, common space areas

One-to-One checks of case number and item numbers shall be conducted when:

- Adding a sample to a container
- Removing a sample from a container and placing it in another container
- Transferring extracted samples for analysis (e.g., TLC plate)

#### **6.3.4.3 SEPARATION**

See *ASCL-DOC-01 Quality Manual*.

### 6.3.5 EXTERNAL ACTIVITIES

---

See *ASCL-DOC-01 Quality Manual*.

## 6.4 EQUIPMENT

### 6.4.1 ACCESS

---

See *ASCL-DOC-01 Quality Manual*.

### 6.4.2 OUTSIDE EQUIPMENT

---

See *ASCL-DOC-01 Quality Manual*.

### 6.4.3 PROPER FUNCTIONING

---

The equipment used in Forensic Chemistry is as follows: microscopes, GCMS, FTIR, balances, reference standards, reference materials (RM), reagents, glassware, solvents, and disposable weighing vessels. All purchased chemicals, reference materials/standards, and disposable equipment are considered fit for use when received. If the packaging is damaged or partially opened, the fitness for use will be assessed. Laboratory equipment and instrumentation shall be handled and transported responsibly to ensure optimal performance and to avoid contamination and premature wear/damage.

The specific maintenance and required performance verification for equipment/instrumentation, to ensure proper functioning and prevent contamination or deterioration, is listed within the test method.

#### OUT OF SERVICE

If an instrument or equipment is not working properly, fails its performance verification, or potential problems are observed, the chemist will immediately take the appropriate steps to repair or correct the problem themselves if they are capable. If the chemist lacks the training or experience to diagnose the problem and restore proper functionality to the equipment, they will clearly mark the equipment 'OUT OF SERVICE' in order to prevent inadvertent use before they seek help in resolving the problem. Maintenance performed to correct the problem must be recorded in the instrument's maintenance log. When it has been determined that instrumentation or equipment was not working properly, the appropriate section chief shall take into consideration the effect the problem may have had on previous tests. Instrumentation or equipment taken out of service will not be used in casework until appropriate calibration or performance verification is performed.

### 6.4.3.1 CHEMICAL, REAGENT, AND REFERENCE MATERIAL RECORDS AND LABELLING

---

#### GENERAL

- All purchased solvents, chemicals, reagents, reference materials shall be marked when received with the date and initials of the person receiving them. Upon opening, the bottle shall be marked with the date and initials of the individual opening the substance.
- The quality of all chemicals purchased for use in the Forensic Chemistry Section will be adequate for their intended use. Generally, this will mean that solvents, acids, bases, organic and inorganic compounds will be of ACS Reagent Grade or better
- Items with a manufacturer-specified expiration date may not be used after that date without documentation to support continued reliability
  - Exception: It is allowable to use hemp reference material as a positive control after the manufacture-specified expiration date if its semi quant response falls in the acceptable range. No certified concentration is provided by the provider for this reference material. The response of this reference material is evaluated with each semi quant batch. Expired reference material will be discarded if its response fails to fall in the acceptable range.
- For items without a manufacturer-specified expiration date, dates will be based on experience, industry standard, or scientific consensus
- Appropriate logs are maintained for reagents/chemicals and reference materials used
- Each analyst must ensure that the reference materials, controls, reagents, or chemicals used in their analysis are of satisfactory quality
- Reference materials, controls, reagents, or chemicals which are determined not to be reliable must be removed from use immediately
- The reliability testing shall occur before use or, if appropriate, concurrent with the test
- Preparation (and verification, if needed) instructions are found on the preparation sheet stored in Qualtrax. Recipes may be scaled up or down depending on need.
- Non-routine reagents may be prepared, if the need arises. The preparation shall be documented to the same degree as the routine reagents on their preparation forms, including verification. This documentation is normally recorded in the case notes, as non-routine reagents shall be discarded after use.
- Glassware used for preparation shall be appropriate and clean. Aqueous preparations shall use distilled or E-pure water.
- Only one batch of each type of prepared reagent/chemical will be in use at a time. A batch's date of initial use is the day after preparation, if the previous batch is still in use, and the date of preparation if not. A batch's date of final use is the earlier of either the batch's expiration date or the date of preparation of the subsequent batch. Before a new batch is put into use, the preparer will notify affected personnel of any excess, from the prior batch, that needs to be discarded. Any exceptions to this will be noted in the appropriate Reagent/Chemical Preparation Book.

## CHEMICALS

**Preparation:** Formulations for preparing routinely used chemicals are located in the *Chemical Preparation Logbook*. Simple solvent mixtures (TLC systems) or acid and base stock solutions will be prepared from materials of adequate quality.

**Verification:** Prepared chemicals (excluding reagents and reference materials) are not normally subject to additional QC measures.

**Labeling:** Containers of chemicals will be labeled with:

- identity
- preparation date (if applicable)
- expiration date (if applicable)

TLC systems will only be labeled with the chemical's identity.

Secondary containers to which purchased chemicals are transferred shall be marked with the identity of the contents and the lot number.

**Documentation:** The *Chemical Preparation Log* must include:

- identity
- preparation instructions
- amount made
- preparation date
- expiration date or expiration time frame
- lot numbers of solvents and/or compounds used in preparation
- initials of the preparer

Controlled forms for all routine chemical preparation logs and recipes are located in Qualtrax.

## REAGENTS

**Preparation:** Formulations for preparing routinely used reagents are located in the *Reagent Preparation Logbook*.

**Verification:** Verification procedures for routinely prepared reagents are located in the *Reagent Preparation Logbook*. Each new batch of reagent that is prepared must be verified prior to use in casework. Verification may be done by the preparer or by another chemist. The verifier will initial the *Reagent Preparation Logbook* for that batch of reagent to certify that the reagent performed as expected.

**Re-verification:** Some reagents may be re-verified. If re-verification is an option, those instructions are located on the reagent preparation form.

**Labeling:** Reagent containers must be labeled with:

- identity
- preparation date
- expiration date

**Documentation:** The *Reagent Preparation Log* must include:

- identity
- preparation instructions
- amount of reagent made
- preparation date
- expiration date or expiration time frame
- lot numbers of solvents and/or compounds used in preparation
- a method to verify the reagent's reliability (if applicable)
- initials of the preparer and verifier of reagent

Controlled forms for all routine reagent preparation logs and recipes are located in Qualtrax.

## REFERENCE MATERIALS

**Verification:** All reference materials (purchased, prepared, secondary, controlled, non-controlled) shall be verified prior to use in casework. The verification can be achieved by a Certificate of Analysis from the vendor or analyzing the sample via FTIR or GCMS. All verification information shall be saved in the Quality Records folder in Qualtrax (Quality Records/Forensic Chemistry/Equipment/Reference Materials).

Secondary Reference Materials (only allowed for qualitative testing)

The collection of reference material from casework shall be documented in the case notes and the agency shall be notified. The secondary reference material shall be recorded in the *Secondary Reference Materials Logbook*; the entry shall include the secondary reference material entry number (SRM#), a unique identifier (e.g., the ASCL case number and item number if it was retained from casework), the method of verification, and the verifier's initials. Containers, for secondary reference materials, shall be labeled with the identity, the secondary reference material entry number, and the unique identifier.

**Preparation:** Prior to preparation, the preparer shall ensure the reference material has proper verification documentation. The preparer shall document the method of preparation on *DRG-FORM-29 Reference Material Preparation*. The procedure description should contain enough detail to ensure reproducibility.

**Labeling:** All prepared reference materials shall be labeled with the reference material's identity, designation, the date of preparation, and the expiration date. Prepared qualitative reference materials expire one year after their preparation or on the date specified on the manufacturers' documents/bottle.

**Reverification:** Prepared qualitative reference materials may be re-verified to confirm the reliability of the reference material. The procedure is listed below:

1. Run reference material on GCMS
2. Both the chromatogram and the mass spectral data shall be evaluated for acceptability for continued use (abundance of the chromatographic peak, presence of any extra peaks that cannot be explained, etc.)
3. Document the reverification and date on the original prep sheet. Record the new reference material designation to include the date of reverification and new expiration date. (e.g., METHA190107 is reverified on 01-09-2020, the new designation would be METHA200109 and the expiration would be 1-9-2021)

All reverification information shall be saved in the Quality Records folder in Qualtrax (Quality Records/Forensic Chemistry/Equipment/Reference Materials) using the naming system: New Designation (reverified from Old Designation). The expiration date shall be set for one year from the re-verification date. In addition to the identity and new reference material designation, the label shall contain the original prep date and the new expiration date.

#### **6.4.3.2 REFERENCE COLLECTION RECORDS**

Forensic Chemistry uses reference collection libraries for comparison to known reference materials in the GCMS, FTIR, and Pharmaceutical Identification testing techniques. Each reference collection has entries documented, uniquely identified, and properly protected.

#### **6.4.4 PERFORMANCE VERIFICATION**

Performance verifications shall occur prior to instrumentation being put into service. These performance verifications shall be documented and retained.

#### **NEW EQUIPMENT PERFORMANCE VERIFICATION**

Performance verification shall be done and recorded prior to putting instruments/equipment into use.

#### **MICROSCOPES**

A plant material sample known to contain cystolithic hairs shall be observed using the new microscope.

#### **GCMS**

After the instrument manufacturer's installation is complete, the following verification shall be performed prior to the instrument being placed into service.

- Add routine methods from instrument with compatible operating software
- Run each routine method with either a test mix of drug reference materials that are commonly identified using that method, or a single drug that is commonly identified with that method

- Evaluate the chromatography, fragmentation patterns, and library matching capabilities to ensure quality performance of the instrument and software
- Retain this data and information in the appropriate location

## FTIR

After the instrument manufacturer’s installation is complete the monthly performance check for the FTIR shall be performed and saved in the appropriate location. If the performance check passes, the FTIR is ready to be put into service.

A performance verification shall be performed on instrumentation and equipment that has gone outside of the direct control of the laboratory (e.g., for repair or preventive maintenance) to ensure that its calibration status is satisfactory before being returned to service. Instrument logs will reflect that the equipment was functioning properly prior to being returned to service.

### 6.4.5 FITNESS FOR SERVICE

---

See *ASCL-DOC-01 Quality Manual*. Also see BALANCES section 9.2 of this document.

### 6.4.6 CALIBRATION REQUIREMENT

---

See *ASCL-DOC-01 Quality Manual*.

### 6.4.7 CALIBRATION PROGRAM

---

#### 6.4.7.1 COMPONENTS

Listed below is the equipment with its calibration interval. The equipment will be calibrated by an ISO/IEC 17025 accredited calibration service provider or replaced after calibration interval has passed.

Calibration certificates shall contain the measurement results, including the measurement uncertainty or a statement of compliance with an identified metrological specification. These certificates will be located in Qualtrax.

Equipment	Calibration Interval	Tolerance ‘as found’
Balances, Analytical	Yearly	1%, safety factor: 2
Balances, Toploading	Yearly	5%, safety factor: 2
Balances, Bulk	Yearly	5%, safety factor: 2
Traceable weights used for performance checks (multiple masses)	10 years	See table below
Electronic Pipettes (300uL, 1000uL)	Yearly	See table below

Nominal Value - Weight	Tolerance 'as found'
10kg	2.0000g
2000g	20.0000mg
100g	0.5000mg
5g	0.0680mg

Nominal volume $\mu\text{L}$ - pipette volume	Maximum permissible error
30 (20-300 $\mu\text{L}$ pipette)	4.0 % (1.2 $\mu\text{L}$ )
150 (20-300 $\mu\text{L}$ pipette)	1.6 % (2.4 $\mu\text{L}$ )
300 (20-300 $\mu\text{L}$ pipette)	0.8 % (2.4 $\mu\text{L}$ )
100 (100-1000 $\mu\text{L}$ pipette)	4.0 % (4.0 $\mu\text{L}$ )
500 (100-1000 $\mu\text{L}$ pipette)	1.6 % (8.0 $\mu\text{L}$ )
1000 (100-1000 $\mu\text{L}$ pipette)	0.8 % (8.0 $\mu\text{L}$ )

The above criteria shall be used to evaluate the pipette's acceptability. If the pipette falls outside of the acceptable range, potential causes shall be evaluated. The pipette shall not be returned to service until it can meet the above criteria.

#### 6.4.8 LABELLING

---

See *ASCL-DOC-01 Quality Manual*.

#### 6.4.9 OUT OF SERVICE

---

See *ASCL-DOC-01 Quality Manual*.

#### 6.4.10 INTERMEDIATE CHECKS

---

Balances and pipettes are subjected to intermediate checks.

#### 6.4.11 CORRECTION FACTORS

---

See *ASCL-DOC-01 Quality Manual*.

#### 6.4.12 EQUIPMENT ADJUSTMENT

---

See *ASCL-DOC-01 Quality Manual*.

### 6.4.13 EQUIPMENT RECORDS

Records are retained for equipment that influences laboratory activities. These records include the Forensic Chemistry Equipment Log, Calibration Certificates, Reagent Logbooks, microscope cleaning/service records, and instrument and balance logs.

## 6.5 METROLOGICAL TRACEABILITY

See *ASCL-DOC-01 Quality Manual*.

## 6.6 EXTERNALLY-PROVIDED PRODUCTS AND SERVICES

If a material or service must meet certain specifications in order to properly function in testing, these items and the required specifications (e.g., manufacturer, type, grade or other technical data relevant to the supply or service) will be communicated to the Procurement Section through a Procurement Request workflow in Qualtrax.

Supplies, reagents, and consumable materials that affect the quality of tests are not used until they have been visually verified to meet the previously-defined specifications. Inconsistencies will be reconciled before materials are utilized in casework.

As chemicals are first opened in the section, the opener is responsible for initialing and dating the container. Supplies, reagents, and consumable materials shall be stored in accordance with the manufacturer's recommendations.

Critical consumables, supplies, and services which affect the quality of testing will be obtained from reliable suppliers.

In the Forensic Chemistry Section, the critical consumables are:

- Certified standards/reference materials
- PFTBA (perfluorotributylamine) GCMS tuning compound (PFTBA does not expire due to the stability of the substance.)

In the Forensic Chemistry Section, the critical supplies are:

- Certified reference weight for performance verification
- Polystyrene reference materials (various forms) for FTIR performance verifications

## 7 PROCESS REQUIREMENTS

### 7.1 REVIEW OF REQUESTS, TENDERS, AND CONTRACTS

#### 7.1.1 GENERAL

---

The Forensic Chemistry Section processes evidence submitted by external law enforcement agencies and the Medical Examiner's Office of the Arkansas State Crime Laboratory. Contracts (submission sheets) are reviewed by Forensic Chemistry personnel to assess the requests made by the customer; if any changes or amendments are necessary all affected personnel shall be notified.

Evidence submitted in death investigation cases is assessed to determine whether the evidence needs processing to aid in the death investigation. The appropriate section chief may contact the agency or the medical examiner of record for more information, should it be needed.

#### 7.1.2 INAPPROPRIATE REQUESTS

---

The Forensic Chemistry Section will not routinely process found property. If a case is identified during submission sheet review to fit this description, personnel should turn the submission form over to the appropriate section chief who may contact the submitting agency to verify that no suspect exists. The section chief, or designee, may then cancel the request for analysis.

Adjudicated cases will not routinely be processed. If the laboratory finds a case closed on the Court Connect website or is notified of a case being adjudicated, then a Drug Send Back Letter will be issued. There is a Drug Send Back Letter for each laboratory location. Send Back Letters may also be issued for cases in which cause and manner of death have been determined and no additional analysis is necessary or for cases in which evidence was found and no suspect is attached to the evidence.

### 7.2 SELECTION, VERIFICATION, AND VALIDATION OF METHODS

#### 7.2.1 SELECTION AND VERIFICATION OF METHODS

---

See *ASCL-DOC-01 Quality Manual*. Forensic Chemistry's test methods are listed in Section 9 of this manual.

#### 7.2.2 VALIDATION OF METHODS

---

See *ASCL-DOC-01 Quality Manual*.

## 7.3 SAMPLING

All evidence sample containers (e.g., test tubes, beakers, auto sampler vials) shall be labeled with at least the significant digits of the ASCL case number and the exhibit (item) number.

When practicable, two separate aliquots from an evidence item shall be collected for analysis using separate analytical testing schemes.

Wet samples shall be allowed to dry before obtaining a net weight and/or sampling.

### 7.3.1 GENERAL

For cases containing multiple exhibits, the chemist will select exhibits to test, based on their training and experience, which will substantiate the highest charge the case will support. Each piece of evidence shall be tested independently<sup>1</sup>. Items listed as probable cause for a search shall be selected for analysis. If there are multiple buys, each buy date shall be tested to the highest charge. Cross contamination of items may preclude the examination of the contaminated items. When practicable, evidence should be left for additional testing, should that be necessary. If the item was a residue or a residual amount is left after obtaining a sample, it is recommended that the GCMS vial be repackaged into the evidence for potential future reanalysis. The case notes shall reflect any inclusion of GCMS vials in repackaging of the evidence and a label notifying the agency shall be affixed to the evidence.

#### SINGLE SUSPECT / MULTIPLE SUSPECTS

Single suspect: If evidence is found on the suspect and in a car, test the highest charge item – if the evidence found on the suspect and the car evidence are equal charges, then test what is on the suspect.

Multiple suspects: Test each suspect to the highest charge and be mindful of what may be attributed to a specific suspect versus to the group.

#### DEATH INVESTIGATION – OVERDOSE CASES

These cases are generally prioritized and processed as received. Testing on these cases does not always follow typical item selection for analysis and may require additional items be tested.

Typically items that are most probative for the death investigation will be tested. Analysts shall consult a section chief or tech lead if guidance is necessary.

#### PRISON CASES

Testing on these cases does not always follow typical item selection for analysis and may require additional items be tested. Typically, all items with different appearances will be tested. Analysts shall consult a section chief or tech lead for guidance if testing requirements become burdensome.

---

<sup>1</sup> One plastic bag twisted into two compartments shall be considered two items for testing and shall be tested separately, if testing is necessary.

## PURPOSE TO DELIVER FACTORS

There are factors that indicate purpose to deliver that shall be considered when processing evidence. The analyst shall consider which items best support the purpose to deliver charge.

Factors that may indicate purpose to deliver as listed in the Arkansas code:

- The person possesses the means to weigh, separate, or package controlled substances
- The controlled substance is separated and packaged in a manner to facilitate delivery
- The person possesses a firearm that is in the immediate physical control of the person at the time of possession of the controlled substance
  - If the AOC CourtConnect record indicates an individual is being charged with a Class Y Felony of simultaneous possession of a firearm and a felony drug charge – the felony controlled substance may be tested to show presence but not up to a weight threshold. The AOC CourtConnect record shall be indexed into the case record.
- The person possesses at least two (2) other controlled substances in any amount

## GUIDANCE ON PURPOSE TO DELIVER

### **Weigh, Separate, Package – Scales**

In deciding what may be necessary to test, scales with plant material residue or no visible residue shall be considered as a D felony, scales with other residue shall be considered as a B felony. Use the “purpose to deliver” column on the Drug Matrix Chart

- Scale and weighable drugs (same charge) – test weighable drugs
- Scale and weighable drugs (lower felony) – test both
- Scale and weighable drugs (misdemeanor) – test scale
- Scale and weighable drugs (higher charge) – test weighable drugs

### **Separated and Packaged**

If there are multiple separated and packaged items (more than one) with the same appearance, this may indicate purpose to deliver. If these separated and packaged items are the basis to show purpose to deliver and substantiate the highest charge, one of these items shall be tested, at minimum.

### **Firearms**

- Can assume possession of a firearm by it being a listed item on the drug submission sheet or a separate submission sheet for firearms
- Summary of crime mentioning a firearm – look it up in AOC CourtConnect to confirm this information and include the AOC CourtConnect information

When considering meeting the three separate controlled substances to show intent to deliver, the drug whose schedule and amount achieves the highest charge will be tested to the maximum

threshold. Two of the remaining types of scheduled drugs will be tested minimally to show presence only.

Exclusions to the three drug rule are as follows:

- Paraphernalia is not considered
- Illicit tablets that are not pharmaceutical mimics will be considered together
- Partial suspected pharmaceutical tablets may be excluded
- Hand rolled cigarettes or cigars (burnt or unburnt) will not be considered unless packaged to demonstrate purpose to deliver
- This is not applicable to federal cases
- A Y Felony has been met
- When multiple exhibits contain a weighable amount of the same controlled substance result

No controlled substances or inconclusive results for tests run on selected items will necessitate the testing of additional items until the highest charge possible is substantiated or the supply of evidence items has been exhausted.

## **7.3.2 SAMPLE SELECTION/COLLECTION**

---

### **7.3.2.1 SOLIDS (PLANT MATERIAL, POWDER/CRYSTALLINE SUBSTANCE)**

Obtain a sufficient portion of the substance to ensure a representative<sup>2</sup> sampling for analytical testing. If the substance is in multiple bags, independently test a portion from enough bags to substantiate the highest charge.

Cigars/cigarettes and butts shall be removed from loose plant material and sub-itemized.

Plants (suspected to be marijuana) are defined as vegetation containing roots, stalks, stems, and leaves. They shall be sampled in the following manner:

1. Note how many plants are present
2. Remove roots
3. Remove small stems and leaves from larger stems and stalk
4. Weigh small stems and leaves
5. Retain a portion for analysis

### **7.3.2.2 MULTI-UNIT POPULATIONS (PHARMACEUTICAL TABLETS, CAPSULES, OR PARTIAL TABLETS)**

The chemist will inspect all the tablets or capsules in an exhibit to ensure consistency and record a count or count by weight, if appropriate, in the case notes. If the item's appearance and imprint code do not indicate the presence of a controlled substance, no further testing is required. Partial

---

<sup>2</sup> Sample from multiple locations throughout a plant sample item. If an item has a mixture of crystal and powder appearances ensure you sample from all or homogenize the sample

tablets can be excluded from testing if they are not suspected to substantiate the highest charge based off of chemist experience.

For items suspected to contain a controlled substance or suspected to be pharmaceutical mimics, a single unit of the population will be tested. The weight of the total population and the weight of the tablet/capsule/partial tablet tested shall be recorded in the case notes and reported.

### **7.3.2.3 MULTI-UNIT ILLICIT TABLETS**

The following scheme shall be followed:

1. Measure, weigh, and assess what may need to be tested
2. Separate, count, and describe groups that need to be tested by shape, color, and imprint
3. Test one tablet, separately, from enough sub items to reach a statutory weight limit based off of the total weight of each sub item OR test one tablet, separately, from a minimum of five sub items.
  - a. If the five tested tablets each contain a controlled substance – the analyst may stop and report their findings after appropriate testing is conducted. The report must contain a note, since testing was truncated (See reporting section for wording).
  - b. If the five tested tablets result in a mix of some with controlled substances and some with only non-controlled substances, or if they are all non-controlled/negative – the analyst shall have the data reviewed and contact the prosecutor about how to proceed.
4. All not tested tablets shall be weighed. Depending on the number of remaining tablet types, the analyst may describe as tablets of various shapes, colors, and imprints or separate by shape, color, and imprint if desired.

### **7.3.2.4 MULTI-UNIT SOLID DOSAGE FORMS (E.G., GUMMY CANDIES, SWEET TARTS, SUGAR CUBES)**

A single unit of the population will be tested. The weight of the total population and the weight of the unit tested shall be recorded in the case notes and reported.

Manufacturer's packaging and serving size may be assessed and it is the analyst's discretion to determine if more than one unit needs to be combined in order to obtain an adequate sample for analysis. If an analyst concludes that more than one unit, up to a serving size, is needed, the number of discrete units (if applicable) and the weight shall be recorded in the notes. When practicable, only a single unit shall be tested.

### **7.3.2.5 MULTI-UNIT POPULATION – CIGARS/CIGARETTES/CIGAR BUTTS/CIGARETTE BUTTS**

The weight of total population and the weight of the unit taken for analysis shall be recorded in the notes and reported.

### **7.3.2.6 SOLID DOSAGE FORMS – MULTI- UNIT OR SINGLE UNIT (E.G., BLOTTER PAPER SQUARES, STRIPS, PAPERS)**

The weight of total population and the weight of the unit/portion taken for analysis shall be recorded in the notes and reported. If multiple portions are taken for analysis, each portion shall be weighed and analyzed separately. The notes shall reflect whether the portion sampled was returned to the case or destroyed/retained/consumed.

### **7.3.2.7 SOLID SINGLE UNITS (E.G., CANDY BARS, LOLLIPOPS, BROWNIES, COOKIES)**

The weight of the solid single unit shall be recorded in the notes and reported. Enough of the solid unit to yield a result may be analyzed. The result from the analysis will be attributed to the whole unit.

### **7.3.2.8 PARAPHERNALIA**

Paraphernalia is not required to be tested unless:

- It is the only evidence in a case (if multiple items of paraphernalia are present the item that substantiates the highest charge, based on chemist experience, shall be tested separately from other pieces; if no controlled substances are detected on the selected piece, testing the remaining pieces separately until a charge is met, or the evidence is exhausted, is required)
- It is probable cause
- It can substantiate the highest charge

The notes must indicate whether there is a residue present or no visible residue. A sample from paraphernalia may be obtained by rinsing the item with a suitable solvent.

### **7.3.2.9 SEEDS**

Seeds are not required to be tested unless:

- They are the only evidence in the case
- They are probable cause
- They can substantiate the highest charge

A sample may be obtained by rinsing the seeds with a suitable solvent, or by crushing the seeds and extracting with a suitable solvent.

### **7.3.2.10 LIQUIDS**

Single layer liquids have a reasonable assumption of homogeneity. If appropriate, agitate the liquid well and transfer a portion directly into a screw top vial or covered test tube in order to avoid evaporation of the sample.

### **7.3.2.11 COMPRESSED ITEMS (PLANT MATERIAL OR POWDERS)**

---

For compressed evidence large enough to conceal items within, a core sample must be taken if the item cannot be broken apart. Notes must reflect if a core sample was taken, otherwise it is assumed it was broken apart.

### **7.3.2.12 STATISTICAL SAMPLING PLAN FOR MULTI-UNIT POPULATIONS**

---

If statistical sampling is desired to result in testing significantly fewer items, the population shall be evaluated for homogeneity. Homogeneity is assessed by comparing all sub-items to each other for visual consistency. If there is a reasonable assumption of homogeneity, a sampling plan may be used. If a sampling plan is employed, the chemist will contact the prosecutor if necessary. The chemist will communicate clearly what will and will not be tested, the inference(s) that may reasonably be drawn from the results, and the manner in which the results will be reported. A section chief or technical leader shall be consulted, prior to sampling, if a calculation to obtain the population net weight will be performed. The purpose for this is to assess homogeneity of the packaged items prior to sampling.

The Forensic Chemistry section employs the Hypergeometric Distribution sampling plan with a confidence level of 95% and a population interval of 90% ( $k = 0.9$ ). Determine the number of sub-items (population) that comprises the exhibit. Consult the hypergeometric calculator spreadsheet (S:\!FC Controlled Information\ENFSI-DWG-Qualitative-Sampling-Calculator.xls) to determine the number of sub-items that must be independently tested. Using the "Hypg\_Proportion" tab in the spreadsheet, only enter your population size in Step 1. Step 2 shall be 0.9, Step 3 shall be 0, and Step 4 shall be 0.95. The analyst shall index the first two pages of the spreadsheet to demonstrate how many items were required to be tested.

#### **Sampling Procedure:**

Randomly select from the population the number of sub-items determined by the sampling plan. For each sub-item, obtain a portion for analysis and perform necessary tests independently on each sub-item. Clearly label the items that were selected for sampling.

If the tested sub-items return non-homogenous results, all remaining items shall be tested independently until a charge is met, or the prosecuting attorney may be contacted to determine what course of action is required for further analysis.

## 7.4 HANDLING OF TEST ITEMS

### 7.4.1 EVIDENCE STORAGE

---

#### 7.4.1.1 HANDLING PROCEDURES

See *ASCL-DOC-01 Quality Manual*.

##### 7.4.1.1.1 STORAGE

Forensic Chemistry has a Secure Storage evidence room within the section in the Little Rock facility. This storage area serves as a temporary location for evidence waiting on processing. Personnel within the Little Rock location have fob access to this storage location.

In the Lowell location, Forensic Chemists have access to the location's Secure Storage due to the size of the laboratory. The Forensic Chemists have access by fob during working hours (8-4:30 weekdays) but need a key for entry outside of those hours.

In Little Rock and Lowell, each analyst has their own personal storage area to secure evidence in their absence. The Little Rock and Lowell locations have storage cabinets to which a key is assigned out to only one chemist at a time when necessary to store bulky items.

If it is necessary to reassign evidence already in an analyst's possession, the section chief may retrieve the evidence from the analyst's storage area(s). The section chief will then reassign and transfer the evidence to another analyst. The chain of custody will reflect all transactions.

The section chief or designee may allow access to an absent analyst's storage area(s) for inventory purposes. The storage area(s) will be locked immediately upon completion of the inventory.

##### 7.4.1.1.2 PACKAGING AND SEALING

If a packaging deficiency is not apparent until the case is checked out by an analyst, the analyst may correct the deficiency. If there is any concern that the packaging deficiency has affected the integrity or identity of the test item, a section chief or technical lead and the customer agency shall be advised and consulted for further instructions. All consultations and remedial actions taken to correct packaging or evidence deficiencies shall be noted in the case record (e.g., submission form or analyst's notes).

It is the responsibility of the analyst to maintain proper control of all evidence in their possession. Evidence may be stored for a short or long period of time in the hood when drying is necessary or when there are safety concerns.

#### 7.4.1.2 CRIME SCENE EVIDENCE (ILLICIT LABS)

---

The following policies and procedures apply exclusively to the Little Rock and Lowell laboratories. The Hope laboratory does not accept evidence associated with controlled substance manufacturing cases (with the exception of marijuana grows).

Illicit Laboratory chemists do not collect evidence from crime scenes for submission to the laboratory. If the ASCL is called to the scene of a clandestine laboratory, our role is to help the on-scene officers know what evidence is appropriate to collect to support manufacturing.

#### 7.4.1.3 RENDERING HAZARDOUS MATERIALS SAFE

---

Evidence Receiving Technicians may call Forensic Chemistry to render materials safe if they are unsure if the packaging is adequate. The Forensic Chemist shall inspect the packaging and repackage any items that need to be rendered safe. This inspection/repackaging shall be documented on *Illicit Laboratory Evidence Safety Form ER-FORM-01*.

Hazardous materials that could be submitted include: organic powders, inorganic powders, organic solvents, strong aqueous bases, strong aqueous acids, pyrophoric<sup>3</sup> metals, noxious gases, flammable vapors, and potent physical drugs (e.g., fentanyl and analogs).

##### 7.4.1.3.1 PACKAGING OF VOLATILE CHEMICALS

---

If the chemical evidence consists of liquids, these liquids should be packed in a glass vial with a Teflon seal, and the glass vial should be placed in a high density non-reactive plastic bottle. Any evidence that emits acidic, basic, organic, or otherwise dangerous fumes that cannot be trapped in the containers specified above shall not be accepted into the Arkansas State Crime Laboratory evidence receiving section.

##### 7.4.1.3.2 PACKAGING OF HAZARDOUS SOLIDS

---

Any solid sample that the chemist determines to have hazardous properties should be placed in a glass vial with a Teflon seal and sealed in a high density non-reactive plastic bottle.

##### 7.4.1.3.3 PACKAGING OF IODINE

---

Iodine shall be packaged with great care to prevent cross contamination. Because of the sublimation properties of iodine, only a small amount (e.g., 1-2 grams) of sample is necessary. It should be packaged in a glass vial with a Teflon seal. The glass vial should then be packaged in a high density non-reactive plastic bottle. Samples of suspected iodine will permeate through most plastic bags and all textile based packaging.

---

<sup>3</sup> Liable to ignite spontaneously on exposure to air

#### 7.4.1.3.4 PACKAGING OF LITHIUM OR SODIUM METAL

Lithium and sodium metal are pyrophoric upon contact with water and should be handled with extreme caution. Lithium or sodium samples should be stored in a heavy organic solvent or petroleum distillate. No alcohol, ether, acetone, or ketone of any kind should be used to store lithium or sodium. A small amount of lithium or sodium (e.g., one inch square) should be placed in a glass vial with a Teflon seal. An organic solvent (e.g., hexanes, petroleum ether, Coleman fuel, camp fuel) should be poured into the glass vial. It is important to keep the surface of the solvent well above the lithium or sodium metal.

#### 7.4.1.3.5 PACKAGING OF ANHYDROUS AMMONIA

Anhydrous ammonia is a very dangerous basic gas. No anhydrous ammonia containers shall be submitted to the Arkansas State Crime Laboratory. If analysis of ammonia is requested, a small amount of ammonia should be bubbled through deionized water. All handling of anhydrous ammonia containers should be done observing safety standards approved by OSHA and the EPA.

#### 7.4.1.3.6 PACKAGING OF SHARPS

Any evidence that is sharp enough to puncture the skin shall be stored in a puncture proof container.

#### 7.4.1.3.7 PACKAGING OF POTENT PHYSICAL DRUGS

Suspected potent drugs shall be packaged in a manner that the drug cannot escape from the packaging.

### 7.4.2 ITEM IDENTIFICATION

---

See *ASCL-DOC-01 Quality Manual*.

### 7.4.3 DEVIATIONS

---

If the analyst discovers an inconsistency between the submitted evidence and the submission sheet, or if there is doubt about the suitability of an evidence item for testing, then the analyst shall consult the customer before proceeding. All contacts will be documented in the case record (e.g., using an *Agency Contact Form* (ASCL-FORM-06), by email). For minor inconsistencies, the analyst shall use their judgment on whether to contact the customer. Plainly visible not listed items do not require agency contact and will not be tested.

Examples that require consultation with the agency:

- Missing evidence
- Item description or agency identifier does not conform to the information provided
  - Green plant material is listed, but crystalline substance was received
  - Extra suspect listed on the packaging
  - Suspect name is substantially different between submission sheet and packaging

- Unexplainable weight discrepancy between the submission sheet and the weight obtained
- Substantial difference in agency case number between submission sheet and packaging
- Hidden evidence not listed on the submission sheet (whether being tested or not)

Should a response not be received within five business days, the evidence may be returned to the investigating agency to correct. Information regarding the reason for return shall be attached to the evidence.

## 7.5 TECHNICAL RECORDS

### 7.5.1 CASE NOTES

The analyst shall create a set of case notes for each case they analyze. The case notes may be handwritten, typed, or a combination of the two. This section outlines what must be included in the case notes. Any deviation from these guidelines must have approval of the section chief or designee.

#### REQUIREMENTS FOR NOTES AND OBSERVATIONS

- Handwritten notes and observations must be in ink. However, pencil may be appropriate for diagrams or making tracings.
- Nothing in the handwritten notes will be obliterated, erased, or deleted. Any corrections made to handwritten notes will be made by an initialed, single strikethrough (so that what is stricken can still be read).
- For training cases, the tasks performed by the trainee or the trainer must be identifiable and clear as to who performed the tasks.
- Each page of the case notes must include:
  - The unique ASCL case number (YYYY-000000)
  - The date(s) the notes were taken on (Should the sampling of a case take longer than one day, it shall be properly noted which day the sampling was resumed.)
  - The handwritten initials of the chemist or trainee (or electronic equivalent). For training cases, if all the work was not done by the trainee, it must be clear which individual was responsible for each activity.
- Every evidence item shall have an adequate description explaining the appearance of the item and its packaging. The description shall be detailed enough so that the chemist could identify the evidence based only on their notes. If evidence is not tested, it must be clearly documented. If there are multiple suspects listed on the submission sheet, any names on inner packaging should be recorded in notes.
- Measurements taken must be documented in the case notes and clearly identifiable to the item to which it corresponds. Brackets shall be placed around the portion of the item and packaging that is included in any gross weights.

- If statistical sampling is employed, it must be clearly documented in the case notes and follow the statistical sampling plan within this manual.
- During testing, the chemist must document in their case record:
  - the tests performed (and by whom, if applicable)
  - the date on which the tests were performed unless supporting examination documentation is marked with the testing date
  - a description and date of any solvent extraction procedure (this excludes tests only for informational purposes like Marquis or Cobalt Thiocyanate to determine possible concentration)
  - the results of the tests (unless the test was non-conforming)
  - non-routine items:
    - re-running a test: how the sample was treated differently, if applicable and not obvious through review of the case record (e.g., concentrated sample down, re-extracted to prepare more concentrated sample, spotted more heavily), or why the sample was re-run (better chromatography)
    - non-conforming results – state why the results are non-conforming (e.g., no test mix run, failed autotune, bad blank, wrong solvent vial run)
    - no controlled substance results – shall provide enough information on extraction method/concentration to ensure enough testing has been conducted to support conclusion
- Any other notations required for an individual testing technique are described within this manual with the test method.
- If the evidence is reopened, this requires documentation in the case notes. The reopen statement shall include the date the evidence was reopened and the purpose for reopening. If this statement is an addition to the case notes, it requires initials and the date of the addition.

#### 7.5.1.1 TECHNICAL RECORD RETENTION

See *ASCL-DOC-01 Quality Manual*.

Sample data from a failed QA/QC sample (e.g., testmix, hemp RM, DP) does not have to be included in the case file. A bad blank from a sample must be included in the casefile if the QA/QC is passing.

Failed controls for SQ (EB, PC, and CBD) and test mix shall be included in the appropriate JT case file.

For Semi-quant, passing DP data does not have to be included in a case file with samples that have a bad blank.

#### 7.5.1.2 ABBREVIATIONS

Each chemist shall use the secure **Forensic Chemistry Abbreviation Definition List** for any abbreviations used in note taking that are specific to the laboratory. This list shall be available to all forensic chemists and can be found on S:\!FC Controlled Information\forensic chemistry

abbreviation definition list. A forensic chemist can add to this list at any time by submitting a new abbreviation to their supervisor or designee. A Forensic Chemistry section chief or designee shall review the abbreviation suggestion for overlap and need before adding it to the secure Forensic Chemistry Abbreviation Definition List. Once the new abbreviation definition is added to the secure Forensic Chemistry Abbreviation Definition List, it may be used by any forensic chemist in their note taking process.

### 7.5.1.3 TECHNICAL RECORD SUFFICIENCY

---

See *ASCL-DOC-01 Quality Manual*.

### 7.5.1.4 TECHNICAL RECORD PERMANENCY

---

See *ASCL-DOC-01 Quality Manual*.

### 7.5.1.5 REJECTION

---

If data, an observation, or a calculation is rejected, the following information will be recorded in the technical record:

- The reason for the rejection
- The identity of the person rejecting
- The date of the rejection

This applies to pre-draft complete items as well as rejections in case review.

Examples of when to reject data include:

- Non-conforming data
  - Bad QA/QC, bad blank
  - DP failed
  - Wrong solvent vial run
  - No RM spotted on TLC plate
  - RM for comparison did not develop color
  - Samples containing compounds with same Rf factor on TLC (e.g., meth/MDMA)
  - TLC spots ran together
  - TLC uneven run - where it is clear samples merged into other lanes (may not have to reject all samples, only the ones affected by the merging of samples into other lanes)
  - Uneven solvent front (may not have to reject all samples, only the ones that appear to be affected)
- Conflicting/inconsistent results
  - Run 1—GCMS: indicative meth, positive  $\Delta 9$ -THC;  
Run 2 (concentrated sample)—GCMS: positive meth,  $\Delta 9$ -THC not detected.  
The result of THC should be questioned and rejected based off the presence of  $\Delta 9$ -THC not being confirmed with a more concentrated sample preparation. If the presence of  $\Delta 9$ -THC

cannot be explained – consult section chief/tech lead as an additional sample may need to be taken to investigate.

- Incorrect results
  - Ran incorrect vial
  - Δ8-THC in 1<sup>st</sup> run that does not persist after liner change (reject Δ8-THC result)
  - Syringe was found to be clogged, and you had sample runs that were blank
  - Psilocin that does not persist with an acetonitrile extraction (reject first run)

## 7.5.2 AMENDMENTS TO TECHNICAL RECORDS

---

Amendments<sup>4</sup> to technical records must be trackable to previous versions or to original observations. Both the original and amended data/files will be retained, including:

- The date of alteration
- An indication of the altered aspect(s)
- The personnel who made the alteration(s)

Any corrections made to existing hardcopy technical records will be made by an initialed and dated single strikeout (so that what is stricken can still be read) by the person making the change. All additions will be initialed and dated. Correction fluid or correction tape may not be used. These corrections may be made to the original document if the original information is still legible.

Contemporaneous<sup>5</sup> revisions to technical records are not considered to be amendments.

### 7.5.2.1 CORRECTIONS REQUIRED AFTER ADMINISTRATIVE REVIEW

---

In instances where a correction or the addition of technical records is required after administrative review, a *Technical Record Addendum* sub-request will be created under the affected request. The Arkansas State Crime Laboratory shall be listed as the requesting agency and “+Case File” shall be listed as the requesting representative. All additional technical records or corrections shall be stored in this request folder. The *Technical Record Addendum* shall be reviewed in the same manner as all technical records with any corrections being documented appropriately.

## 7.6 EVALUATION OF MEASUREMENT UNCERTAINTY

The Forensic Chemistry Section has measurement of uncertainty estimates for weight determination on the analytical, toploading, and bulk balances (Little Rock and Lowell)

---

<sup>4</sup> Including additions, deletions, changes, interlineations, or any other modification to the original information

<sup>5</sup> Contemporaneous means at the same period of time. Amendments made after moving on to the next case are not considered to be contemporaneous. Amendments made before moving on to the next case, while the matter is still fresh in memory, may be considered contemporaneous.

The budgets are reviewed/recalculated on a yearly basis, at minimum, and when there are significant changes to procedures or equipment has undergone calibration. The budget for weighing devices shall be updated and/or recalculated when new equipment is put into service.

The current revision of MU budgets is retained in Qualtrax and shall be used. The requirements for measurement uncertainty reporting are addressed separately within this manual.

## 7.7 ENSURING THE VALIDITY OF RESULTS

### 7.7.1.1 VERIFICATION

The Forensic Chemistry Section does not perform verification of independent examinations.

### 7.7.1.2 CASE REVIEW

All cases will be technically and administratively reviewed prior to the release of the report. The review process must confirm that electronic versions of all necessary documentation are in the imaging module of the JusticeTrax program. Each technical and administrative review will cover, at minimum, the items listed on *ASCL-FORM-05*.

If a reviewer discovers an error in the case record, the reviewer must document the error, their initials, and the date in the *Reviewer Notes* field, in the related request in JusticeTrax, and inform the analyst. If the analyst and reviewer cannot reach a consensus, then both the analyst and reviewer must meet with a section chief or the technical lead for resolution.

If the error requires the analyst to correct administrative and/or examination records, the correction may be made to the original record, if appropriate, or the original record will remain in the electronic case file and the corrected record stored with a different name (e.g., corrected notes, corrected data). If there is a change to the report, the original report shall be added into the JusticeTrax case file and should be marked (e.g., "Draft Report", "Original Report").

Sometimes bigger cases will require two reviewers. Once each reviewer has completed the review, they will meet and record the errors, their initials, and the date in the *Reviewer Notes* field in JusticeTrax and inform the analyst. The completion of the review should be recorded by one reviewer rolling the tech review milestone and the other reviewer rolling the administrative review milestone.

#### 7.7.1.2.1 TECHNICAL REVIEW

See *ASCL-DOC-01 Quality Manual*.

#### 7.7.1.2.2 ADMINISTRATIVE REVIEW

See *ASCL-DOC-01 Quality Manual*.

### 7.7.1.2.3 TESTIMONY REVIEW

The Forensic Chemistry Section ensures that analysts are accompanied and reviewed by a competency tested and authorized Forensic Chemist on their first testimony. Should improvements be needed, they may be re-reviewed on future testimonies to ensure they are effective. For all other requirements, see *ASCL-DOC-01 Quality Manual*.

---

## 7.7.2 INTERLABORATORY COMPARISONS

See *ASCL-DOC-01 Quality Manual*.

---

## 7.7.3 MONITORING ACTIVITY ANALYSIS

See *ASCL-DOC-01 Quality Manual*.

---

## 7.7.4 INDIVIDUAL PERFORMANCE MONITORING

See *ASCL-DOC-01 Quality Manual*.

---

## 7.7.5 PERFORMANCE MONITORING REQUIREMENTS

The Forensic Chemistry Section follows the policy in *ASCL-DOC-01 Quality Manual* for proficiency testing/intralaboratory comparisons. Test specimens may be obtained from external providers or prepared internally. Intralaboratory comparisons may include previous proficiency test samples, samples retained from casework (secondary proficiency reference materials), samples prepared from primary reference materials, re-examination techniques, and blind techniques.

The Forensic Chemistry Section uses the following criteria to evaluate the results of proficiency tests/intralaboratory comparisons.

### MASS DETERMINATION EVALUATION

A mass determination component may be included as a part of a proficiency test/intralaboratory comparison. Tests that include this component are determined prior to assignment.

**SATISFACTORY:** Mass range is  $\pm 5$ mg from preparation measurement. Any sample loss (e.g., static, transfer loss) may cause variation from this range.

Section chief evaluation is required if reported measurement is outside of the expected range.

**UNSATISFACTORY:** Mass range is outside of the expected range with no justification.

### QUALITATIVE PROFICIENCY TEST / INTRALABORATORY COMPARISON EVALUATION

**SATISFACTORY:** Identification of all expected controlled substances and general chemicals/non-controlled substances

UNSATISFACTORY: Incorrect or incomplete identification of expected controlled substances and general chemicals/non-controlled substances

## CLANDESTINE LABORATORY ANALYSIS EVALUATION

SATISFACTORY: Correct identification of all controlled substances, non-controlled substances, and/or elements expected

UNSATISFACTORY: Incorrect or incomplete identification of all controlled substances, non-controlled substances, and/or elements expected

### 7.7.6 PERFORMANCE MONITORING SCHEDULE

---

The proficiency testing/intralaboratory comparison schedule is maintained by the Section Chief and/or the Technical Leader and is available on Qualtrax.

### 7.7.7 PROFICIENCY TEST SOURCING

---

See *ASCL-DOC-01 Quality Manual*.

### 7.7.8 PERFORMANCE MONITORING RECORDS

---

See *ASCL-DOC-01 Quality Manual*.

### 7.7.9 RE-EXAMINATION POLICY

---

The Forensic Chemistry casework re-examination policy is a quality assurance policy that is intended to evaluate the reliability of our analytical results and the competency of our analysts. This is achieved by internal comparison of results for cases chosen for re-examination.

Re-examination shall be performed by a second competent analyst. Only the items originally analyzed will be re-analyzed. The evidence will be re-examined using appropriate procedures and test methods.

All re-examination data, notes, and other documentation shall be contained within the original case record under the "Re-examination Request." The original analyst's name (Last, First) shall be recorded in the "Requester" box located on the Request Tab in JusticeTrax.

Results of both testing activities shall be compared by the analyst performing the re-analysis. Any discrepancies shall be evaluated. Discrepancies that cannot be explained shall require investigation by the Chief Forensic Chemist and/or Technical Leader.

While the main focus of casework re-examination is the comparison of original analytical result(s) to re-examination results, the re-examination should also include the evaluation of some, or all, of the following:

- that the original descriptions were adequate and complete

- that all seals made by the original analyst are appropriate and properly labeled
- that the item(s) and outer packaging were properly sealed to prevent contamination or deleterious change
- that all counts and weights are consistent with original documentation
- if applicable, that the analyst correctly selected items to test to substantiate the potential highest charge based on the appearance of the items and/or additional information listed on the inner packaging (e.g., evidence listed with a specific suspect's name or date of offense/buy date)
- if applicable, that the notes reflect any repackaging and/or inclusion of GCMS vials

All conflicting results and/or observations shall be evaluated to determine the level of significance of the discrepancy. If necessary, a Quality Assurance Concern (QAC) shall be initiated.

Any nonconformities, deficiencies, or departures from accepted laboratory or section standards identified during the course of re-examination shall be documented and may require initiation of a QAC. If a QAC isn't required, the documentation will be maintained by the Chief Forensic Chemist and/or Technical Leader. Due to the wide variability of Drug casework and potential occurrences necessitating action, a different degree of response may be required from one instance to another. If retraining or remedial actions are necessary, an action plan will be developed based on the nature of the cause. Actions taken may include, but are not limited to, observation-based casework monitoring, removal of authorities/responsibilities, re-examination of additional casework, and/or other actions as deemed necessary.

Cases undergoing re-examination, as part of the quality assurance program, shall have a re-examination letter issued. If a change was necessitated, the letter shall state "This case was selected for re-examination as part of our quality assurance program. This re-examination necessitated a change to the original report of analysis; an amended report will be issued." If no change was necessitated, the letter shall state "This case was selected for re-examination as part of our quality assurance program. This re-examination did not necessitate a change to the original report of analysis; no additional report will be issued."

An amended report is necessary when:

- original reported results are incorrect
- there is an error on the original report that needs correction

## 7.8 LANGUAGE FOR REPORTS AND TESTIMONY

### 7.8.1 GENERAL

---

See *ASCL-DOC-01 Quality Manual*.

### 7.8.1.1 DOCUMENTATION

---

See *ASCL-DOC-01 Quality Manual*.

### 7.8.1.2 REPORTS

---

An ASCL “Report of Laboratory Analysis” is generated at the conclusion of analytical testing. These reports normally consist of administrative information in a “header” and technical information in the report body. For each item listed the report body contains two columns of information: “Items” and “Evidence Description”. When applicable, the body may also include statements about evidence sampling or disclaimers that aid the customer in understanding the report.

If there were multiple submissions, on the same case, from separate investigating officers, a report shall be issued to each officer containing the evidence they submitted for analysis.

The results of testing carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively. This document describes general guidelines intended to cover reporting of the majority of cases analyzed. However, situations may arise which require deviation from these guidelines due to the extreme variability of evidence received. In such a case, the chemist will consult the section chief to determine an approved method for reporting the information.

Upon completion of the report, the chemist shall review the header information (e.g., investigating officer/agency/address, suspect(s), victim(s), laboratory case number, agency case number) and the analytical results (item numbers, item descriptions, test results, weights, etc.) reported to ensure they are correct. Once proofing is complete, the analyst will sign the report by marking the request ‘Draft Complete’ in JusticeTrax.

## 7.8.2 COMMON REQUIREMENTS FOR REPORTS

---

### 7.8.2.1 REPORT ELEMENTS

---

For a broad list of elements included and not included in the report see *ASCL-DOC-01 Quality Manual*.

#### 7.8.2.1.1.1 GUIDELINES FOR ALL ITEMS RECEIVED

---

All items (exhibits) submitted, shall be included on the report.

#### ITEMS LISTED ON SUBMISSION SHEET

The following shall be present for all reported items:

- The item (exhibit) number
- A description of the item including, where appropriate, count/count by weight
  - Containers are not required in report descriptions with the exceptions of:

- Items where reported gross weight includes any packaging. Reported description shall reflect all items included to obtain gross weight or a note shall be present to make it clear what the reported gross weight reflects.
  - Paraphernalia (smoking device, grinder, syringe, vape pen/cartridge, etc.) containing weighable substances
- The exhibit's net weight with units and measurement uncertainty or gross weight with units (in grams) with exceptions of:
  - Items for which a weight is not required to be taken by this manual
  - Items that contained a residue or no visible residue: report residue, where appropriate
  - Items that weigh less than 50mg: report as "less than 50mg" with no measurement uncertainty
  - Non-controlled pharmaceutically identified tablets
  - Factory cigarettes/cigars free of suspected tampering/alteration (count only)
- The result(s) of analytical testing, if it was conducted (see guidelines for reporting items that were tested)
- The result of "not tested" for items not undergoing testing. For multi-unit populations the "not tested" can be a part of the evidence description.
- Any appropriate disclaimers:
  - Color-tested - only items or other partial testing
  - Chemist assignment of item numbers with reason
  - Relatable discrepancy in item number(s)
  - Notification that debris could not be removed
  - For items where pharmaceutical testing results will be reported: Pharmaceutical Identification disclaimer (should auto populate when results are entered into Justice Trax)
  - For items pharmaceutically identified to contain a controlled substance, items that were indicative or negative for pharmaceutical identification, or non-controlled items with positive pharmaceutical identification where no "identified as\*" result was reported, where no other analysis was conducted, the following disclaimer shall be on the report. "Tablets were compared to reference sources."
  - Truncated Testing disclaimers for illicit tablet testing
  - Not Listed/Not Tested disclaimers
  - Semi-Quant disclaimers (should auto populate when results are entered into Justice Trax)
  - Illicit Lab disclaimer, if needed (e.g., ammonia, lithium, sodium, and certain metals)
  - A disclaimer to clarify the report or help the customer to understand may be necessary in certain circumstances

## ITEMS (OF EVIDENTIARY VALUE) NOT LISTED ON THE SUBMISSION SHEET

Not listed items shall be listed at the bottom of the report. No weight is required to be reported on these items (but it must be recorded in the notes).

Example: NOTE: “The following items were not listed on the submission form and received no analysis: one straw, one plastic bag, and one plastic bag containing green vegetable material.”

#### 7.8.2.1.1.2 GUIDELINES FOR ITEMS THAT WERE TESTED

---

##### GENERAL

- Salt/base form or diastereomer may be reported based on a positive FTIR test
- If testing positively identifies a set of stereoisomers in an item but does not positively identify the specific stereoisomer present, the results may be reported as *stereoisomer a/ stereoisomer b* (e.g., pseudoephedrine/ephedrine, zopiclone/eszopiclone, citalopram/escitalopram)
- Items that break down during testing to another substance that is the same schedule/charge or items that cannot be distinguished between with our testing shall be reported as follows:
  - psilocyn/psilocybin
  - gamma-butyrolactone (GBL)/gamma-hydroxybutyric acid (GHB)
- If no element, compound, or substance was positively identified, the results may be reported as “no controlled substances detected”
- When practicable, significant (either in abundance or relevance) non-controlled drug/common cutting agent peaks shall be reported (e.g., large non-controlled substance in the presence of a controlled substance, non-controlled drug with no controlled substance, smaller xylazine peak with fentanyl, small nitazene peak with large syn-can peak)
- When practicable, all significant (either in abundance or relevance) controlled drugs shall be confirmed and reported, even when other controlled drug(s) substantiate a higher charge in that item (e.g., a large meth peak with a smaller fentanyl peak, a large syn-can peak with a nitazene compound)
- CBD shall be reported when it is present in items with the exception of those in which marijuana is reported
- For items that were **color tested only**, the following disclaimer (or a rewording with similar language to include multiple items/tablets/capsules) shall be on the report: “A color test was performed to screen for the presence of various organic functional groups”

## TABLETS / CAPSULES AND SEALED PHARMACEUTICAL PRODUCTS

- Both count and weight shall be reported for tablets or capsules tested or identified to contain a controlled substance
- For items pharmaceutically identified to contain non-controlled substances that receive no further analysis, the active ingredients, and if desired the dosage, may be reported.  
If only non-controlled substances are present in the case, the pharmaceutical identification shall be reported. The result shall be preceded by the phrase “identified as\*” and the report shall contain the following disclaimer “\*The identification results were obtained by comparing the item to reference sources and not by analytical testing. Any results confirmed by analytical testing are listed separately.”
- For items pharmaceutically identified to contain a controlled substance that breaks down on the GCMS to another drug, the active ingredients, and dosage (if desired), shall be reported if the substance substantiates the highest charge or is the third drug to show purpose to deliver. The result shall be preceded by the phrase “identified as\*” and the report shall contain the following disclaimer “\*The identification results were obtained by comparing the item to reference sources and not by analytical testing. Any results confirmed by analytical testing are listed separately.”
- For items pharmaceutically identified to contain a controlled substance, items that were indicative or negative for pharmaceutical identification, or non-controlled items with positive pharmaceutical identification where no “identified as\*” result was reported, where no other analysis was conducted, the following disclaimer shall be on the report. “Tablets were compared to reference sources.”

## MULTI-UNIT POPULATIONS

The report must state what was submitted, what was tested, and must be clear that the result/conclusion pertains only to what was tested. Illicit tablet cases in which testing was truncated without contacting the prosecutor shall contain the following note. "If more testing is necessary for this case, please contact a Drug Section Chief and your request for additional testing will be prioritized."

- For multi-unit populations composed of solid dosage forms (e.g., LSD on blotter paper squares, gummies, tablets, partial tablets, capsules, sublingual films)
  - A description (which includes the word "submitted") of the entire population including a gross weight/net weight and identity of dosage form (if known)
  - A description (which includes the word "tested") of what was tested, the total net/gross weight of the items tested, and the results
  - If more than one discrete unit was combined in order to obtain an adequate sample for analysis, a note shall be added to the report making it clear that the units were combined.
- For all other multi-unit populations, when reported together and only a portion of the population is tested, the report may include in some variation:
  - A description (which includes the word "submitted") of the entire population including a gross weight/net weight
  - A description (which includes the word "tested") of what was tested, the total net weight of the items tested, and the results
  - A description (which includes the words "not tested") of what was not tested, the total gross weight/net weight of the items not tested

## SEMI-QUANTITATIVE RESULTS FOR PLANT MATERIAL TESTING

### **Plant material with response ratio greater than or equal to 1 and criteria for reporting marijuana have been met:**

- Report the appropriate result in the JusticeTrax module to display the desired result and the following disclaimer. <sup>1</sup> "As determined by comparison with a 1%  $\Delta$ 9-tetrahydrocannabinol standard." If "marihuana" is reported, no other significant cannabinoids should be reported for clarity in reporting.

### **Plant material with response ratio less than 1 and criteria for reporting $\Delta$ 9-THC have been met:**

- Report the appropriate result in the JusticeTrax module to display the desired result and the following disclaimer. <sup>2</sup> "The response of this sample was less than that of a 1%  $\Delta$ 9-tetrahydrocannabinol standard. If quantitation is necessary, please contact the laboratory for further analysis."

**Plant material with a negative microscopic test with response ratio greater than or equal to 1 and criteria for reporting Δ9-THC have been met:**

- Report the appropriate result in the JusticeTrax module to display the desired result and the following disclaimer. <sup>3</sup> “The response of this sample was greater than or equal to that of a 1% Δ9-tetrahydrocannabinol standard.”

**MANUFACTURING CASES**

- If only elements or non-controlled substances were positively identified in an exhibit, the chemist may report “no controlled substances detected” with or without the positively identified elements or substances
- The following table outlines the proper method to report results which may be significant in the manufacturing process.

<i>Reporting Results Significant to Manufacturing Cases</i>	
<b>Result Reported</b>	<b>Necessary Tests</b>
Phosphorus/Iodine	XRF
Inorganic salts (e.g., Ammonium nitrate, Sodium phosphate)	IR solid
Ammonia <sup>1</sup>	IR vapor, Nessler’s
Lithium <sup>2</sup>	IR solid, Flame Test
Lithium metal	IR solid, Flame Test, Reactive w/ H <sub>2</sub> O
Sodium <sup>3</sup>	XRF, IR solid
Sodium metal	XRF, Reactive with H <sub>2</sub> O
Other elements <sup>4</sup>	XRF alone, or IR and Flame Test
Acidic solution/basic solution	pH test
<p><sup>1</sup> The following disclaimer must be added between the report’s last evidence item and the analyst’s signature: “The presence of ammonia does not confirm the presence of anhydrous ammonia.”</p> <p><sup>2</sup> The following disclaimer must be added between the report’s last evidence item and the analyst’s signature: “The presence of lithium does not confirm the presence of elemental lithium.”</p> <p><sup>3</sup> The following disclaimer must be added between the report’s last evidence item and the analyst’s signature: “The presence of sodium does not confirm the presence of elemental sodium.”</p> <p><sup>4</sup> The following disclaimer must be added between the report’s last evidence item and the analyst’s signature: “The presence of “<i>insert element</i>” does not confirm the presence of elemental “<i>insert element</i>”.”</p>	

**7.8.2.1.1.3 GUIDELINES FOR ITEMS THAT WERE NOT TESTED**

The report shall clearly communicate which items (or portions of items) were not tested.

## 7.8.3 SPECIFIC REQUIREMENT FOR TEST REPORTS

---

### 7.8.3.1 ADDITIONAL STATEMENTS

See *ASCL-DOC-01 Quality Manual*.

### 7.8.3.2 REPORTING SAMPLING

If the Hypergeometric Distribution sampling plan is used, the report for the item must also include a statement indicating the confidence level and population interval specified by the plan. See the example below for guidance on reporting.

Example (change item number and individual units as needed): “A hypergeometric distribution sampling plan was used for the analysis of E1. Twenty-eight (28) units were selected at random. The results of the individual testing of those units were consistent. At a confidence level of 95%, it can be statistically stated that 90% of the submitted units contain the reported result.”

## 7.8.4 SPECIFIC REQUIREMENTS FOR CALIBRATION CERTIFICATES

---

See *ASCL-DOC-01 Quality Manual*.

## 7.8.5 REPORTING SAMPLING – SPECIFIC REQUIREMENTS

---

Please refer to Section 7.8.3.2.

## 7.8.6 REPORTING STATEMENTS OF CONFORMITY

---

See *ASCL-DOC-01 Quality Manual*.

## 7.8.7 REPORTING OPINIONS AND INTERPRETATIONS

---

See *ASCL-DOC-01 Quality Manual*.

## 7.8.8 AMENDMENTS TO REPORTS

---

### 7.8.8.1 IDENTIFYING THE CHANGE(S)

An amended report is necessary if an error is found on an issued report (including reports uploaded to JusticeTrax iResults portal). An “amended request” will be created in JusticeTrax and all administrative and examination records for the amended analysis will be added to the electronic case record. Administrative and technical reviews are required before an amended report is issued. The Section Chief or Technical Lead will administratively review all amended requests. If the amended report is necessitated due to an error missed by the original reviewer, they may be included in the amended review process. Should two reviewers be needed, documentation of this review will be incorporated into the original case file, by each reviewer rolling one of the

milestones on the Amended Request. When an amended report is necessitated by a change in analytical results, then the Section Chief or Technical Leader will perform both the technical and administrative review on the amended request.

The original report and all original records will be kept in the case record.

When an amended report is issued, any change of information will be clearly identified. Where appropriate, the reason for the change will be included on the report.

### **7.8.8.2 STYLE OF AMENDMENT**

---

See *ASCL-DOC-01 Quality Manual*.

### **7.8.8.3 IDENTIFYING THE AMENDED REPORT**

---

The statement “AMENDED REPORT: This corrected report replaces the report dated [DATE]” (or equivalent) will appear below the header information and above the listing of the evidence and the results. The date of the original report must be entered in the “additional data” tab of the amended request. The amended report will contain all of the items on the original report and any amendments.

### **7.8.9 SUPPLEMENTAL REPORTS**

---

A supplemental report is necessary when additional evidence is received after the original report has been issued, additional requests for analysis are made, or other additional testing is required in a case (Note: When additional evidence is received on a case that has not been completed, the additional evidence may be analyzed and included in the original report.) The date of the original report must be entered in the “additional data” tab of the supplemental request.

Evidence resubmitted to the laboratory for testing shall be inspected by the analyst to ensure it is in substantially the same condition as when the analyst completed the original analysis. Items not undergoing additional testing may be described only. If the case has left the control of the laboratory, a new weight shall be recorded in the notes, for items undergoing additional testing, regardless of whether it is necessary for reporting. If the analysis associated with a supplemental request is completed by the original chemist, original weights must be used on the supplemental report. Only items undergoing additional testing will be included in the supplemental report.

If the analysis associated with a supplemental request is completed by a chemist other than the one assigned to the original request, a supplemental report is necessary if **ONLY** new items are tested.

If reanalysis of previously tested items occurs (this excludes the quality assurance re-examination program), the request type shall be a retesting drugs request. The date and request number of the original report must be entered in the “additional data” tab of the retesting drugs request. The retesting request may also include new tested items and amendments to the original report. When

an amendment is required, any change of information will be clearly identified on the retesting drugs report. Where appropriate, the reason for the change will be included on the report.

## **7.8.10 RETESTING REPORTS**

---

See *ASCL-DOC-01 Quality Manual*.

## **7.8.11 LANGUAGE FOR TESTIMONY**

---

Forensic Chemistry analysts may be called to testify to their conclusions in a court of law. The analyst shall testify without bias and stay within the boundaries of their area of expertise.

An analyst may testify to any of the following:

- Education, training, experience
- Results of analytical testing
- Conclusions from analysis
- Weight of sample and associated measurement of uncertainty
- Analytical testing scheme and theory of how the tests work

### **7.8.11.1 DEFINITIONS OF CONCLUSIONS**

---

#### **IDENTIFICATION OF SUBSTANCE THROUGH ANALYTICAL TESTING**

The substance has been identified through appropriate analytical testing. The analyst may testify to the conclusion of the substance being present in the sample analyzed.

#### **IDENTIFICATION OF SUBSTANCE THROUGH PHARMACEUTICAL IDENTIFICATION**

Analyst may testify that physical characteristics and imprint on the submitted evidence were consistent with the pharmaceutical reference source. They may make no assertions as to what is actually in the item unless separate analytical testing was conducted.

#### **NOT IDENTIFIED (NO CONTROLLED SUBSTANCES DETECTED)**

Analyst may testify that we were unable to detect or confirm any controlled substances through the analytical testing performed.

**SEMI-QUANT RESULTS** (This testing is indicated on the report of analysis by superscript 1,2, or 3 in front of the reported results and should contain a disclaimer with the same number)

Marihuana – Analyst may testify that the sample is marihuana if the appropriate testing scheme has been followed (See 9.1.1.2). The analyst may state whether the response of the sample was greater than or equal to the response of the 1% Δ9-THC standard. The analyst may not make any statement on the potential Δ9-THC concentration in the sample.

$\Delta$ 9-THC – Analyst may testify to the components detected in the sample if the appropriate testing scheme has been followed (See 9.1.1.2). The analyst may state whether the response of the sample was greater than, less than, or equal to the response of the 1%  $\Delta$ 9-THC standard. The analyst may not make any statement on the potential  $\Delta$ 9-THC concentration in the sample.

## MANUFACTURING

An Illicit Lab chemist can testify to substances that meet the requirements for reporting. They may provide their opinion on the manufacturing method.

Analysts may not testify to manufacturing in plant material cases.

### 7.8.11.2 LIMITATIONS OF DRUG ANALYST TESTIMONY

- If a substance is identified in casework evidence, the analyst shall make no assumption or suggestion as to the source of the evidence, how that substance was transferred to the evidence, or how long that substance has been present in the evidence.
- When analyzing a portion of a population, an analyst shall not state their conclusion applies to the entire population (or a percentage of the population) unless statistical sampling was employed. When statistical sampling is employed, the analyst shall clearly explain the conclusion being made, the results of the sampling units tested, and the confidence level.<sup>6</sup>
- An analyst shall not state that drug chemistry examinations are infallible or have a zero error rate.
- An analyst shall not provide a conclusion that includes a statistic or numerical degree of probability except when based on relevant and appropriate data.
- An analyst shall not cite the number of drug cases worked in their career as a direct measure for the accuracy of a proffered conclusion. They may cite the number of cases worked in their career for the purpose of establishing, defending, or describing their qualifications or experience.
- An analyst shall not use the expressions “reasonable degree of scientific certainty,” “reasonable scientific certainty,” or similar assertions of reasonable certainty in either reports or testimony

## 7.9 COMPLAINTS

See *ASCL-DOC-01 Quality Manual*.

---

<sup>6</sup> When hypergeometric distribution sampling is conducted, the analyst can testify to the visual consistency of the sub items within the population. The analyst can testify to the number of randomly selected and independently tested sub-items and their individual results. The analyst must communicate at a confidence level of 95% that 90% of the sub-items statistically contain the reported substance.

## 7.10 NONCONFORMING WORK

See *ASCL-DOC-01 Quality Manual*. The Chief Forensic Chemist retains records of simple corrections for the section.

## 7.11 CONTROL OF DATA AND INFORMATION MANAGEMENT

See *ASCL-DOC-01 Quality Manual*.

## 8 MANAGEMENT SYSTEM REQUIREMENTS

See *ASCL-DOC-01 Quality Manual*.

## 9 TEST METHODS

This section describes the testing techniques commonly utilized for the analysis of evidence exhibits in the Forensic Chemistry Section.

If it becomes necessary to make a deviation from a documented method or procedure, it must be technically justified and authorized by the appropriate section chief. The deviation will be documented in the case record.

If a prosecutor needs to be contacted on how to proceed based on preliminary results, the analyst shall have the data reviewed by another competent analyst before contacting the prosecutor. Documentation of the data review shall be included in the case file.

### 9.1 TESTING REQUIREMENTS

Minimum testing requirements are listed within this section. Categories of testing are used to address a number of the minimum testing requirements. The categories of common testing techniques are listed in the table below.

<i>Categories for Common Testing Techniques</i>		
<b>Category A</b>	<b>Category B</b>	<b>Category C</b>
Infrared Spectroscopy (IR), Gas Chromatography/Mass Spectrometry (GCMS), Energy Dispersive X-Ray Fluorescence (EDXRF)	Gas Chromatography (GC), Thin-Layer Chromatography (TLC), Microscopic Examination	Color Tests, Pharmaceutical Identifiers, pH

If conflicting test results are obtained during the course of testing, this shall be evaluated. Evaluation may trigger re-running of already performed tests or taking another aliquot of sample to confirm previously obtained test results. If the analyst cannot determine the reason for the conflicting results, a section chief or the technical leader shall be consulted.

#### 9.1.1 MINIMUM TESTING PER EXHIBIT

For items selected for analysis, at a minimum, two tests per item must be performed in order to report *Test Results* for that item on the *Report of Laboratory Analysis* generated at the conclusion of testing<sup>7</sup>. Minimum testing for identification of compounds and exceptions are addressed below.

Newly encountered or unfamiliar substances shall be evaluated to determine if the proper reference material needs to be procured to confirm the presence of the substance.

<sup>7</sup> In order to report results other than “not tested,” “*element(s)*” name, “acidic solution/basic solution,” or “identified as *drug*” on the *Report of Laboratory Analysis*, the minimum testing requirements per exhibit listed in this section must be met.

#### 9.1.1.1 MINIMUM TESTING FOR IDENTIFICATION OF CONTROLLED/PENALTY COMPOUNDS (EXCLUDING ITEMS SUSPECTED TO CONTAIN Δ9-THC/CBD)

For each controlled compound identified in an item, the analyst shall have, at a minimum, two positive tests for that compound. One of these tests must be from category 'A'. If only two tests are performed, the Category 'A' test shall be a GCMS with broad temperature program, unless there is substantial reason for choosing a shorter method (e.g., suspected mushroom material, suspected LSD sugar cubes). The second test may be from Category 'A' or 'B', but not Category 'C'.

There are exceptions to this requirement for some pharmaceutically identified tablets/capsules.

Reasons for identification only with no analytical testing include:

- Tablets were identified to contain a drug that does not substantiate the highest charge
- Tablets were identified to contain a substance that breaks down on the GCMS to another drug or breaks down completely (e.g., clorazepate, modafinil, chlordiazepoxide)

Reasons for identification with some analytical testing include:

- Analytical testing was performed on a tablet or capsule and the GCMS data showed that the substance broke down, either completely or into another drug; the result shall be reported as "identified as\*"
- Tablets or capsules where the substance does not chromatograph well using both gas chromatography and thin layer chromatography; the pharmaceutical identification may be used as the second test with a positive GCMS (e.g., pregabalin)

During the course of minimum testing, if results indicate that a substance contains more than one controlled substance there is no obligation to exhaustively confirm the presence of all the controlled substances present. At a minimum, the drug that results in the highest penalty level must be confirmed. If more than one drug satisfies the highest penalty level requirement the chemist may look at additional factors (e.g., availability of reference material, suitable test methods) when selecting which compound to identify.

#### FENTANYL AND FENTANYL ANALOGS

Due to the nature and low concentration of fentanyl and fentanyl analogs commonly found in samples, it is not uncommon that the molecular ion is unable to be obtained. Some fentanyl and fentanyl analogs have very similar mass spectra that make them difficult to distinguish without the acceptable molecular ion present (usually M-2). When any fentanyl or fentanyl analog is detected

in a sample and it does not contain the acceptable molecular ion, the sample shall be confirmed using GCRT with the fentanyl method or appropriate default method.

### 9.1.1.2 MINIMUM TESTING FOR ITEMS SUSPECTED TO CONTAIN Δ9-THC OR CBD

---

#### CASES CONTAINING A FELONY AMOUNT<sup>8</sup> OF LOOSE<sup>9</sup> PLANT MATERIAL THAT SUBSTANTIATES THE HIGHEST CHARGE

##### POSITIVE MARIHUANA IDENTIFICATION REQUIREMENTS

1. Positive microscopic test for cystolithic hairs
2. GCMS semi-quant with Δ9-THC response ratio greater than or equal to 1, where a positive Δ9-THC spectrum was obtained
3. One of the following:
  - a. Positive thin-layer chromatography for Δ9-THC
  - b. Qualitative gas chromatography for Δ9-THC – this must be done if there are interfering compounds identified via GCMS analysis that exceed the threshold<sup>10</sup> to use TLC (The GCRT shall be run on a method with broad temperature parameters, if thin layer chromatography with iodoplatinate development is not performed, to ensure other controlled compounds are not present.)
  - c. Modified Duquenois-Levine color test (with no other cannabinoids present and a broad temperature program was used on GCMS)

#### PLANT MATERIAL THAT IS A MISDEMEANOR AND SUBSTANTIATES THE HIGHEST CHARGE, IS PROBABLE CAUSE, OR POTENTIAL THIRD DRUG<sup>11</sup>, AND OTHER MATERIALS SUSPECTED TO CONTAIN CONTROLLED CANNABINOIDS/CBD (OILS, RESIDUES, FOOD, WAXES, VAPE CARTRIDGES)

Positive controlled cannabinoid identification requires a positive result from Gas Chromatography-Mass Spectrometry (GC-MS) analysis and a positive from any one of the following tests:

---

<sup>8</sup> Dispensary cases may have less than a felony weight submitted but still require testing for identification of marihuana

<sup>9</sup> “loose” means not contained in paraphernalia or hand rolled cigarettes/cigars unless packaged to demonstrate purpose to deliver

<sup>10</sup> See Section 9.6.1

<sup>11</sup> “third drug” excludes hand rolled cigarettes or cigars (burnt or unburnt) unless packaged to demonstrate purpose to deliver

- Thin-layer chromatography when no interfering compounds have been identified via GCMS that exceed the threshold<sup>12</sup>
- Modified Duquenois-Levine (for cannabinoids) (with no other cannabinoids present)
- Qualitative gas chromatography (positive retention time match for cannabinoid of interest) (The GCRT shall be run on a method with broad temperature parameters to ensure other controlled compounds are not present if the GCMS screen was run on a short method and no TLC with iodoplatinate development was performed.)

### 9.1.1.3 MINIMUM TESTING FOR IDENTIFICATION OF ELEMENTS & NON-CONTROLLED COMPOUNDS

For items where only an element or non-controlled compound is identified, the analyst shall have, at a minimum, one positive category 'A' test and a second test from category 'A' or 'B'. If only two tests are performed, the Category 'A' test shall be a GCMS with broad temperature program, unless there is substantial reason for choosing a shorter method (e.g., suspected mushroom material, suspected LSD sugar cubes). The exceptions to this requirement are:

1. The analyst may reach a positive conclusion on the presence and identity of elements in an exhibit based on results from either of these testing schemes:
  - X-Ray Fluorescence testing
  - A positive FTIR result and a flame test consistent with that element
2. The analyst may reach a conclusion that inorganic salts (e.g., ammonium nitrate, sodium phosphate) are present based off of a positive FTIR result.
3. The analyst may reach a conclusion that lithium is present in an exhibit based on the following tests:
  - a positive result for lithium hydroxide or lithium carbonate by FTIR testing
  - results of a flame test consistent with lithium
4. The analyst may reach a positive conclusion to the identity of a non-controlled substance based off of a positive pharmaceutical identification.
5. Federal List I & II chemicals shall require two positive tests to report a result except those that cannot be identified by GCMS (e.g., methylamine).
6. The analyst may reach a conclusion that ammonia is present in an exhibit based on the following tests:
  - a positive result for ammonia by FTIR testing
  - results of a Nessler's color test consistent with ammonia

### 9.1.1.4 ADDITIONAL TESTING REQUIREMENTS FOR ISOMERS AND SALT/BASE FORM

If differentiation between diastereomers (e.g., pseudoephedrine and ephedrine) is desired, or salt/base determination is necessary, infrared spectroscopy testing must be conducted. The IR

---

<sup>12</sup> See Section 9.6.1

results must be positive to report the diastereomer or salt/base form. FTIR is not required for reporting if a positive pharmaceutical identification of a stereoisomer was obtained.

Federal sentencing specifies different penalties for cocaine base and cocaine hydrochloride. For cases being federally prosecuted, weighable items in which cocaine is detected must be analyzed by infrared spectroscopy, so that, if possible, the cocaine form may be determined for reporting.

### 9.1.1.5 TAMPERING ANALYSIS

The Forensic Chemistry section tests suspected tampering cases for the presence of drugs only.

### 9.1.1.6 MONEY

If money is discovered in a case, the analyst will take an inventory of the money and record it in their notes. If money is to be tested for the presence of drug residue, the analyst will do a money shake and test the residue acquired from the shake. Money received as evidence shall not be rinsed.

## 9.2 WEIGHT MEASUREMENT

### 9.2.1 SCOPE

Determination of the weight of solids/powders/tablets, liquids, plant material, etc. Mass may be determined on one of three balances: analytical, toploading, or bulk. Weight measurements shall be made in grams and recorded in the case notes. Weighing shall be carried out on a performance checked balance appropriate for the sample. Reusable weighing vessels shall have a solvent rinse obtained prior to introducing sample into the weighing vessel. These rinses may serve as the solvent blank for the sample if the same solvent is used for sample extraction.

The balance weight ranges are listed in the table below. The uncertainty for reporting and the budgets for uncertainty are located in Qualtrax. When a new version of the uncertainty budget is published, the uncertainty values on that budget shall go into effect the following calendar day for newly reported weight measurements. (For supplemental and amended reports, the uncertainty in effect at the time of the initial report will be used for previously weighed and reported items)

Balance	Weighing Range	Uncertainty of Measurement for a Single Weighing
Analytical	0.0500g-100.0000g	see current budget in Qualtrax
Toploading	5.0g –3000.0g	see current budget in Qualtrax
Bulk (LR/LWL)	400g – 32,000g	see current budget in Qualtrax

Weights recorded in case notes are assumed to be net unless otherwise designated. It is the responsibility of the analyst working the case to clearly label gross weights, calculated net weights,

or counts by weight in their notes. Items that are normally consumed (edibles, sublingual films, capsules, etc.) are considered a net weight.

## 9.2.2 REAGENTS/STANDARDS/CONTROLS

---

- 5g, 100g, 2000g, 10kg reference standard weights

## 9.2.3 SAMPLE PREPARATION

---

### TESTED ITEMS

All items that will be tested shall have their initial net weight measured unless the item is a residue. Chemists shall make every reasonable attempt to remove foreign material mixed in with evidence items. If it cannot all be reasonably removed the notes shall state, why and the chemist shall try and remove enough clean sample to reach a weight threshold for testing and gross weight the remaining substance and foreign material. If no separation can reasonably be achieved, the entire item shall be reported as a gross weight. The notes shall state why separation was not achievable and the report shall be clear the foreign material was not separated.

Exceptions to this requirement are:

- Tablets/capsules pharmaceutically identified to contain no controlled substance may be counted only
- Sealed packages (controlled and non-controlled) may be counted only, but if testing of a unit/tablet occurs, it shall have a net weight taken
- Evidence in manufacturing/tampering cases may not be weighed based off of the chemist's training and experience
- Samples that are color tested, with no further analysis, may have a gross weight measured
- A sample that cannot be readily separated from its container may have a gross weight measured. The notes shall indicate the sample could not be removed from the container.
- Liquid in paraphernalia or liquid that was clearly marked to have come from paraphernalia (syringe liquid, bong water, etc.)
- Vape cartridges do not require a weight measurement be taken
- Gross weight shall be recorded for paper/patches used to administer drugs and not typically consumed

A reserve weight shall be measured after a portion is taken for analysis. Exceptions for the reserve weight are:

- Situations where the sample mass is negligible compared to the mass of the item, the mass of the sample may be satisfactorily substituted
- Samples that are color tested only
- Patches or papers that have been soaked where a portion was not sampled for analysis

## NOT TESTED ITEMS

All not tested items shall have a net or gross weight measured unless the item is a residue. Items with foreign material mixed in, not undergoing testing, shall be reported as a gross weight and it is assumed no attempt was made to remove the foreign material since the item was not tested.

Exceptions to this requirement are:

- Items that are excluded from weight requirement, if tested, are also excluded from weight requirement if they are not tested
- Evidence in manufacturing/tampering cases may not be weighed based off of the chemist's training and experience
- Factory cigarettes/cigars free of suspected tampering/alteration (count only)
- Items that have no evidentiary value (count only)

When multi-unit populations are processed and not all of the units are tested, multiple weighings are required. It is the responsibility of the chemist to ensure all weights are clearly labelled and there is no ambiguity to the relationship between the measurement and the items measured.

## COUNT BY WEIGHT

For exhibits containing more than 160 tablets or capsules that are consistent in size/shape/markings across the group the analyst may calculate the total number of tablets or capsules present in the exhibit using the following equation. The count by weight shall be truncated to a whole number.

$$n_{calc} = \frac{m_{total} \times n_{count}}{m_{count}}$$

Where  $n_{calc}$  = total number of tablets/capsules calculated;  $m_{Total}$  = total mass of the tablets/capsules measured;  $n_{Count} \geq 160$ , the number of tablets/capsules counted; and  $m_{Count}$  = the mass of  $n_{Count}$ .

Example: The chemist receives exhibit E1 which is a small pail filled with several thousand blue tablets. An inspection shows that all the tablets are the same size and inscribed with the same markings. All the tablets together have a weight of 241.8136 grams ( $m_{Total}$ ). 160 tablets ( $n_{Count}$ ) are counted out and are measured to have a weight of 10.2121 grams ( $m_{Count}$ ).

The chemist calculates the total number of tablets ( $n_{calc}$ ):

$$n_{calc} = (241.8136 \times 160) / (10.2121) = 3788.66 \text{ tablets}$$

and truncates the answer to report 3788 tablets **by weight**.

## TOTAL NET WEIGHT AND MEASUREMENT UNCERTAINTY

For a single exhibit containing multiple items or consecutive multiple exhibits with identical reported test results and weighed on the same balance, the individual weights as recorded may be summed. The uncertainty for the summed weights depends on the number of measurements taken

(n) and the uncertainty associated with each weighing operation (u). The reported uncertainty can be calculated using the equation below. The resulting uncertainty shall be rounded up not truncated. Pre-calculated uncertainties for **n** values up to 50 can be found on the Summary sheet of the uncertainty spreadsheet *DRG-DOC-04 MU Budgets*.

<p style="text-align: center;"><b>Calculating MU for Multiple Weighings</b></p> $U = n \times u$ <p><i>U</i> = total uncertainty, <i>n</i> = number of weighings, <i>u</i> = uncertainty in a single weighing</p>
---

## CALCULATED NET WEIGHTS AND MEASUREMENT UNCERTAINTY

When multi-item populations are sampled and a conclusion may be inferred about the whole population, the net weight of the entire population will be calculated from measurements on the item and sub-items using the “Estimation of WEIGHT” tab of the ENSFI-DWG-Qualitative Sampling Calculator. (S:\!FC Controlled Information\ENFSI-DWG-Qualitative-Sampling-Calculator.xls)

Step 1: Enter 0.95 for 95% confidence interval

Step 2: Enter the total number of items in the population

Step 3: Enter the number of items you are sampling and weighing

Step 4: Enter the mean of the weights collected (the individual weights should be entered into an excel file to facilitate review)

Step 5: Enter the standard deviation for the mean weight (this should be performed on excel as well)

Step 6: Enter 0, as we will allow no negative results

Step 7: Enter the uncertainty for one weighing event on the balance used

The calculated net weight for the population “Estimate of Weight” and the calculated uncertainty are highlighted in yellow on the spreadsheet.

### 9.2.4 QUALITY ASSURANCE/CONTROL MEASURES

Balances may require daily and monthly performance checks. Checks shall be performed with NIST-certified weights.

#### MAINTENANCE

- **Daily:** clean and level the balance

Other maintenance is done on an as-needed basis and recorded in the balance log.

## PERFORMANCE VERIFICATION

<b><i>Routine Daily Balance Checks</i></b>	
<b>Daily Checks</b>	<b>Actions</b>
Is the balance level?	Level the balance
Is the balance clean?	Clean the balance
Has the balance been performance checked?	Weigh and record verification weight
Was the balance within tolerance?*	If no, perform adjustment before use If yes, Balance ready to use
* Analytical balance – 100g ± 0.0005g, 5g ± 0.0002g, Toploading balance – 100g ± 0.1g, 2,000 ± 0.1g Bulk balance – 10,000g ± 2g, 2000g ± 1g	

Each chemist's balance(s) will be subjected to the performance checks in the table above on a daily basis before use. The results of the checks and the serial number (or identifying number) of the calibrated weight used for the checks will be recorded on the appropriate log sheet *DRG-FORM-02, 03, 04, 05*. The performance check tolerances will be at least double the tolerance listed on the calibration certificate, but may be administratively set higher.

If a check measurement is significantly out of tolerance (double the allowable tolerance or higher) a Section Chief or Technical Lead shall be consulted prior to any actions being performed on the balance (e.g., moving, leveling, or adjusting the balance). The balance is not performing correctly and must be assessed while the out of tolerance measurement can still be observed by a Section Chief or Technical Lead.

If the balance fails performance checks or if it is not in tolerance after it has been adjusted, the balance must be removed from service for repair. If a balance needs more than two adjustments per performance check event, a Section Chief or Technical Lead shall be consulted. After the balance has been repaired, the balance must be leveled and performance checked before it is returned to service. All repairs, maintenance, and standard weights used must be documented on the appropriate log sheet. If a balance is moved out of the allowed footprint of the balance, re-calibration is necessary before returning to service.

If a balance is transferred to a separate laboratory location, the balance shall be calibrated prior to being put into service.

## 9.2.5 INTERPRETATION OF RESULTS

---

### 9.2.5.1 PRECAUTIONS/POSSIBLE SOURCES OF ERROR

---

- Place items to be weighed on center of balance
- Make sure the weighing area is free of air drafts
- Use clean and tared weighing vessel
- Balances needs to be on a sturdy surface
- Wear gloves to protect the integrity of the reference standards

### 9.2.5.2 POSSIBLE SOURCES OF ERROR INCLUDE BUT ARE NOT LIMITED

TO:

- 
- Static interference
  - Failure to tare or improper taring of the weighing vessel
  - Portion of the weighing vessel not on the balance
  - Balance will not stabilize

## 9.2.6 DOCUMENTATION REQUIREMENTS

---

The weight shall be recorded in case notes. If the initial weight is calculated in some manner, all weighings shall be documented in the case notes and how the calculation was performed shall be clear.

If the chemist must use a balance other than their personal issue (e.g., another chemist's balance or the bulk balance), it is the responsibility of the chemist using the balance to determine whether the required performance checks have been performed that day. If the balance has not been checked, the required performance checks must be performed and recorded before the balance may be used in casework. The chemist shall indicate which balance was used when weighing(s) were done on a balance not assigned to them.

## 9.3 GAS CHROMATOGRAPHY–MASS SPECTROMETRY (GCMS)

### 9.3.1 SCOPE

---

This test method is a two-part test used to screen a wide range of substances in which the gas chromatography portion separates components in a mixture, and the mass spectrometry portion detects the components.

### GAS CHROMATOGRAPHY PARAMETERS

Instrument operation parameters are only one factor in obtaining good separation. A wide variety of parameters may be adjusted by the Forensic Chemist with many combinations of parameters

producing acceptable separation. The chemist should rely on their education and training concerning the theoretical and practical aspects of gas chromatography in the selection of instrumental parameters. A separation in the resulting chromatogram should be evaluated by the chemist on the basis of efficiency (the narrowness of the peaks), the peak shapes (e.g., whether they tail or front) and the resolution represented.

Some of the acquisition conditions that the chemist may adjust are parameters such as: injection volumes, injector mode (e.g., split, splitless), temperature [e.g., of the inlet or oven (initial and final, ramps)], and flow rates. Regardless of the actual instrumental conditions the chemist uses, those conditions must be documented so that the resulting data could be reproduced if necessary.

Instrument parameters shall be in the case record, either on the chromatographic data or included separately. If included separately from the chromatographic data, the method shall be identifiable to the sample for which it was used.

## MASS SPECTROMETER PARAMETERS

Qualitative data shall always be collected in full scan mode with the high mass scanned exceeding the analyte's molecular weight by at least 10 amu.

### 9.3.2 REAGENTS, STANDARDS AND CONTROLS

---

Controls and standards used within this test method are described within their portion of the quality assurance/control portion of this test method.

### 9.3.3 SAMPLE PREPARATION AND ACQUISITION

---

The sample may be prepared by a solvent dilution or extraction and should not be acidic or basic or contain any solid material. One or more blank(s) shall be prepared to appropriately assess the chemical(s), solvent(s), and reusable extraction equipment (e.g., beakers, separatory funnels, mortar and pestle). One prepared blank shall be run immediately before the sample with the same acquisition parameters.

The acquisition parameters shall be chosen based on screen testing, chemist's experience, or tablet identification. A broad temperature ramp is required for samples previously run on a shortened method with no controlled substance results.

GCMS casework blank vial positions are as follows:

1-5: Methanol

26-30: Methylene Chloride or base extraction using methylene chloride

51-55: Other (e.g., ACN, CH<sub>2</sub>Cl<sub>2</sub> with drops of MeOH, Pet Ether, MeOH dried down and then base extracted)

76-80: Reusable Equipment Blanks (81-85 may be used if additional spots are necessary)

For automated injections, the sample and blank are each placed in auto sampler vials and capped. An aliquot of the sample may either be automatically or manually injected into the instrument using a syringe.

### 9.3.4 QUALITY ASSURANCE/CONTROL MEASURES

---

#### MAINTENANCE

- **As Needed:** change septum and liner (non-SQ sequences: as soon as practical after 500 shots, SQ sequences: before each batch), air and water check (recommended after septum and liner change), rinse and refill solvent vials (daily), rinse waste vials (daily), change filaments, clean source, ballast pump
- **Recommended monthly:** Change solvent/waste vials, clean needle guide, clean inlet cap
- **Recommended yearly:** Change rough pump oil, clean inlet, replace gold seal, replace split vent filter, clean fans
- Other maintenance will be performed when the instrument's performance deems maintenance necessary. Maintenance shall be recorded on the GCMS maintenance Log.

#### *PERFORMANCE VERIFICATION*

- **Monthly:** autotune

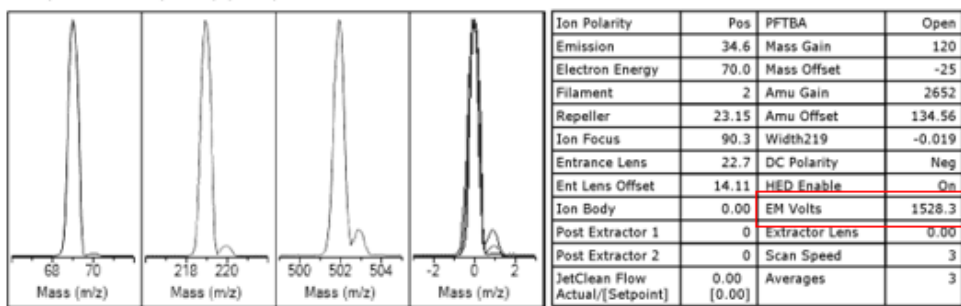
Autotune uses PFTBA (Perfluorotributylamine) masses 69, 219, and 502 to optimize and adjust various parameters for the Mass Selective Detector (MSD). A report is generated (FIG. 1). Autotune must be performed at least once a month. Autotunes shall be indexed into the designated ASCL case number for the instrument and recorded on *DRG-FORM-08*.

Autotunes may be performed more frequently if necessary.

### Autotune - 5977

Tune timestamp: 7/22/2024 8:10 AM (UTC-05:00)  
 D:\MASSHUNTER\GCMS\1\5977\ATUNE.U

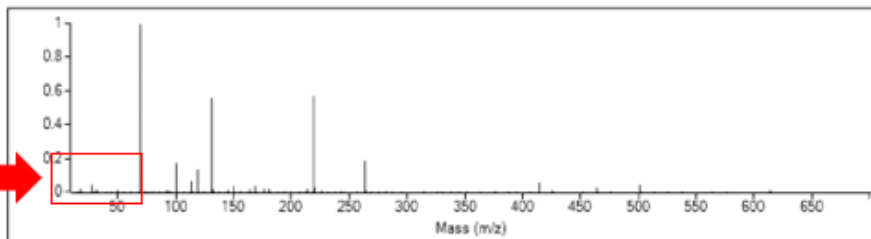
GCMS #2  
 US1917P008



Actual m/z	Abund	Rel Abund	Pw50
69.00	371,878	100.0%	0.59
218.90	211,667	56.9%	0.59
501.90	16,009	4.3%	0.58

Temperatures and Pressures		
MS Source	230 Foreline	50.306
MS Quad	150 Hi Vac	7.63e-05

Low	High	Step	Speed	Threshold	Peaks	Base	Abundance	Total Ion
10.00	701.00	0.10	3	100	122	69.00	356,736	1,163,390



Target m/z	Actual m/z	Abund	Rel Abund	Iso m/z	Iso Abund	Iso Ratio
69.00	69.00	356,736	100.0%	70.00	3,754	1.1%
219.00	219.00	204,096	57.2%	220.00	8,862	4.3%
502.00	502.00	14,925	4.2%	503.00	1,354	9.1%

Air/Water Check: H2O ~1.5% N2 ~4.2% O2 ~0.7% CO2 ~0.4% N2/H2O ~274.2%  
 Column(1) Flow: 2.00 Column(2): 0.00 ml/min Interface Temp: 280

**Ramp Criteria:**

Ion Focus maximum 90 volts using ion 502; Electron Multiplier Gain 60057.334  
 Repeller maximum 35 volts using ion 219; Gain Factor 0.6006

Mass Gain Values(Scan Speed): 134(3) 137(2) 158(1) 185(0) 238(FS1) 290(FS2)

TARGET MASS:	50	69	131	219	414	502	1050
Amu Offset	134.6	134.6	134.6	134.6	134.6	134.6	134.6
Entrance Lens Offset	14.1	14.1	14.1	14.1	14.1	14.1	14.1

### Important Areas of the MSD Report

The chemist must evaluate the performance check by examining the labeled areas of the report for the following conditions:

**1: EM voltage < 2500**

**2: The Actual m/z** (mass assignments) for the tune masses should be within +/- 0.2 m/z of the actual mass at m/z 69.0, 219.0, and 502.0. (68.8 to 69.2, 218.8 to 219.2, and 501.8 to 502.2)

**3:** The **PW50** (Mass Peak Width at 50% Height) of all three tune masses should be  $0.60 \pm 0.05$   $m/z$  (0.55 to 0.65  $m/z$ )

**4: Abundance of any peak(s) below 69  $m/z$**  (e.g., 18[water], 28[nitrogen], 32[oxygen]) should be < 20%, with relative to the abundance of the peak at mass 69,

**5: Isotope Ratio (Iso Ratio)** – should be close to the theoretical values ( $m/z$  70 at 1.08%,  $m/z$  220 at 4.32% and  $m/z$  503 at 10.09%),  $\pm 20\%$  (0.9 to 1.3%, 3.4 to 5.2%, and 8.1 to 12.1%)

If any of these conditions do not pass, then the instrument is not in proper working condition and shall be removed from service until it has been repaired and has passed a performance check.

▪ **Daily prior to use: Test Mix**

Test Mix: Analytes in a mixture to include methamphetamine, cocaine, a benzodiazepine, and opiate.

- a. The mixture will be evaluated for acceptable chromatographic peak shapes, abundance of analytes, and positive mass spectrum matches for all analytes
- b. The Test Mix data shall be indexed into the ASCL case number for the instrument and marked “Pass” or “Fail” on *DRG-FORM-08*.

If substantial changes (e.g., absence of an analyte, significant changes in abundance, increased need for subtraction) occur, notify the appropriate personnel. The instrument should be taken out of service until the issue is resolved and the instrument passes performance checks. If a SQ sequence is the only thing run on an instrument, a test mix does not need to be run because positive controls and reference materials are run with the sequence to determine if the instrument is functioning properly.

▪ **Daily for casework sequences: Sequence Verification**

Another individual shall check the sequence for the following items and initial *DRG-FORM-08*.

- a. Case number and item number
- b. Vial numbers
- c. Methods for blank and sample
- d. Data path and sample name

During the process of loading, the analyst should check the log’s previous lines to ensure that the proper documentation for QA/QC has been recorded. If a sequence has been found to be unverified, the analyst shall notify the Chief Forensic Chemist or Technical Leader.

**As Needed:** For non-SQ samples, a cannabidiol (CBD) or other appropriate reference material shall bracket samples for GCRT testing when an analyte of interest is at a low abundance and its presence could be due to conversion (examples listed below):

- CBD has a greater abundance than  $\Delta 9$ -THC on GCMS – analyte of interest  $\Delta 9$ -THC
- CBD has a greater abundance than  $\Delta 9$ -THC and  $\Delta 8$ -THC on GCMS – analyte(s) of interest  $\Delta 9$ -THC and/or  $\Delta 8$ -THC
- there is no CBD and a greater abundance of  $\Delta 9$ -THC than  $\Delta 8$ -THC – analytes of interest  $\Delta 9$ -THC and  $\Delta 8$ -THC

## 9.3.5 INTERPRETATION OF RESULTS AND REQUIRED DOCUMENTATION

---

### 9.3.5.1 PRECAUTIONS TO BE TAKEN

#### INSTRUMENT:

- Performance check(s)
- Syringe or column blockage

#### SAMPLE PREPARATION:

- Poor choice of solvent (low analyte solubility) or extraction scheme
- Sample concentration is too dilute
- Insufficient sample taken for analysis
- The sample and reference material should not be acidic or basic or contain any solid material

#### RUNNING THE SAMPLE:

- Co-eluting compounds
- Improper acquisition parameters

### 9.3.5.2 POSSIBLE SOURCES OF ERROR

It is the chemist's responsibility to evaluate the chromatogram for any significant peaks. Possible sources of error include:

- Not evaluating all chromatographic peaks
- Co-eluting compounds that create difficulty in identifying substances
- Unsuitable acquisition parameters
- Compounds with similar mass spectrums
- Liner induced compound breakdown (e.g.,  $\Delta 9$ -THC to  $\Delta 8$ -THC, 1P-LSD to LSD)
- Extraction induced conversion (e.g., 4-acetoxy DMT to psilocin)
- Heat-induced conversion (e.g., NBOH compounds)

### 9.3.5.3 CRITERIA FOR POSITIVE, NEGATIVE, AND INDICATIVE RESULTS

---

The sample shall be evaluated for chromatographic peaks. The evaluation of the total ion chromatogram shall include, but isn't limited to peak shape and peak signal to noise.

Criteria for evaluation of chromatographic peaks:

- Blank: any peak with a signal to noise  $\geq 5$  should be evaluated
- Sample: any peak with a signal to noise  $\geq 10$  should be evaluated, exceptions may occur based on analyst's discretion (e.g., weak sample prep, suspected compound that does not chromatograph well)

Once a peak has been determined to need evaluation, the analyst will obtain the mass spectrum and should evaluate the fragmentation pattern and fragment ratios prior to comparison to a reference material spectrum.

An acceptable blank will not contain any peaks (signal to noise  $\geq 5$ ) whose mass spectrum is a positive or indicative match for a controlled substance, drug, or common cutting agent.

Identification of unknown compound(s) in a sample is based on comparing the sample's mass spectrum to reference spectra. Reference spectra can come from a library, literature, or otherwise-known spectrum. Software matching algorithms are useful for rapidly narrowing the number of possible matches, but ultimate responsibility rests with the chemist to determine whether a sample's mass spectrum matches a given reference spectrum.

Subtractions are permissible provided that the chemist includes the following data in the case file:

- A printout of the original full mass scan for the signal area
- A printout of the full mass spectrum that is being subtracted
- A printout of the subtraction results

The electronic data files generated from each GCMS run (samples and blanks) will be retained, at a minimum, until the case is both technically and administratively reviewed.

#### 9.3.5.3.1 POSITIVE RESULTS

---

The mass spectrum, of a peak in the sample's chromatogram, is visually similar to that of the reference material spectrum and the following criteria are met:

- The signal-to-noise ratio of the chromatographic peak is  $\geq 15$
- The blank meets acceptable criteria
- If the reference spectrum contains a molecular-ion peak for the compound, the sample's mass spectrum must also contain the molecular ion peak
  - Exception for aliphatic amines: – These compounds do not have a strong molecular ion peak and often the M-1 ion is more attainable. For aliphatic amines, the M-1 peak is acceptable for making a positive identification (aliphatic amine examples: methamphetamine, amphetamine, MDMA, MDA).

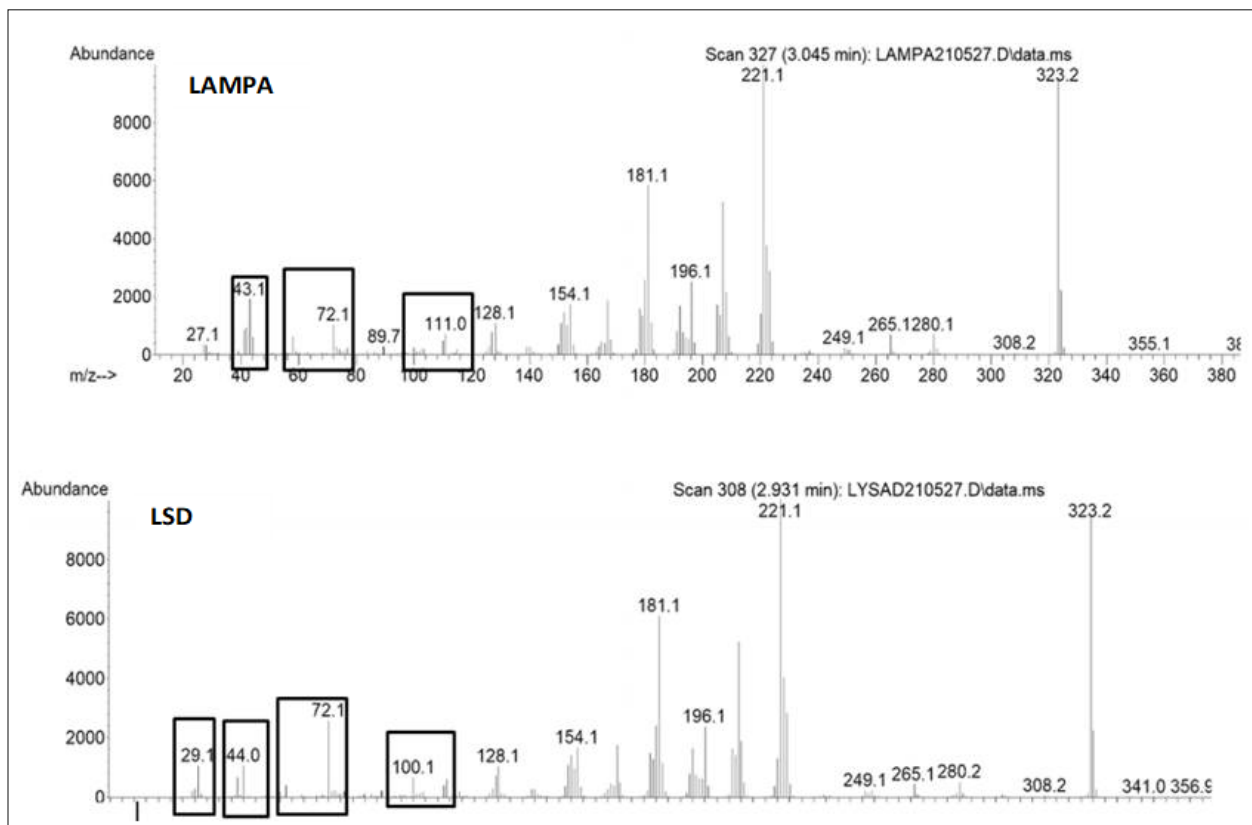
- Exception for compounds where the molecular ion is difficult to obtain: ephedrine/pseudoephedrine, diphenhydramine, fentanyl, fentanyl analogs, and BTMPS
- All peaks present in the reference spectrum shall be present in the sample's spectrum with the following exceptions:
  - Peaks in the reference spectrum that are below the scan limits set in the method parameters [**NOTE:** the appropriate method shall be used for samples suspected to contain compounds such as GBL, GHB, and pregabalin, and LSD which have significant peaks outside the normal scan limits]
  - Peaks in the reference spectrum that are higher in mass than the molecular-ion or the molecular-ion isotopic peaks (if applicable)
  - Low abundance ions (below 10% of the abundance of the base peak) may be absent unless the ion is also the molecular ion
- There shall not be any extra peaks in the sample's spectrum when compared to the reference spectrum with the following exceptions:
  - Low background peaks (below 10% of the abundance of the base peak) are ignored
  - If the reference spectrum has a limited scan range, the sample spectrum shall be compared to a different reference or a reference material spectrum can be acquired for comparison

Other things to consider

- The ion ratios in the sample and reference material are reasonably consistent (this is important in the following scenarios - cannabinoids, cathinones, synthetic cannabinoids, and LSD/LAMPA)

Distinguishing between LSD and LAMPA – see table and mass spectra below  
(Samples must be run on LSD method so that it scans low enough to detect 29 fragment)

LAMPA	LSD
29 fragment (likely present but not significant)	29 fragment (significant fragment, should be greater than 10% base peak)
44 fragment < 43 fragment	44 fragment > 43 fragment
58:72 ratio (72 fragment is roughly double)	58: 72 ratio (72 fragment is significantly larger)
100 fragment < 110/111 fragments	100 fragment $\approx$ 110/111 fragments



### 9.3.5.3.2 INDICATIVE RESULTS

---

The following qualify a sample to be considered indicative:

- The signal-to-noise ratio of the chromatographic peak is  $\geq 10$
- The mass spectrum of a peak in the sample's chromatogram is visually similar to that of the reference material spectrum, but the sample's mass spectrum doesn't meet all of the criteria for a positive result

### 9.3.5.3.3 NEGATIVE RESULTS

---

The mass spectrum of a peak in the sample's chromatogram does not visually match any available reference material spectra or the sample does not meet the signal to noise requirement to be indicative or positive.

## 9.3.6 REQUIRED DOCUMENTATION FOR GCMS RESULTS

---

### 9.3.6.1 GENERAL

---

For exhibits subjected to more than one GCMS test, the chemist will develop a way to relate the sample preparation/test results in the case notes to the corresponding instrumental printout(s).

#### Instrumental Printouts

Images of chromatograms and mass spectra (blanks, reference materials, and samples) supporting the analyst's conclusions must be incorporated into the electronic case file (e.g., by scanning or printing to the JusticeTrax Indexer program) before the case request status is marked 'Draft Complete' in JusticeTrax. The unique ASCL case number, exhibit number, vial number, and date must be visible on the image. For runs utilizing a nonstandard method, a copy of the instrumental parameters (method) must also be incorporated into the electronic case file.

### 9.3.6.2 POSITIVE RESULTS

---

Any compound(s) meeting the criteria for positive results may be entered into the case notes by chemical name or an appropriate abbreviation. Controlled substances meeting the criteria for positive results must be entered in the case notes by chemical name or an appropriate abbreviation<sup>13</sup>. If no controlled substances are present, other significant substances (either in abundance or relevance) must be entered into the case notes by chemical name or appropriate abbreviation.

---

<sup>13</sup> Known breakdown products and manufacturing byproducts that are controlled are excluded from this requirement. Controlled precursors are excluded from this requirement unless they are the only controlled substance detected or the highest charge.

### 9.3.6.3 INDICATIVE RESULTS

---

Any compound(s) meeting the criteria for indicative results may be entered into the case notes by chemical name or an appropriate abbreviation followed by a question mark and the notation will be enclosed in parentheses. Controlled substances meeting the criteria for indicative results must be entered in the case notes by chemical name or an appropriate abbreviation.<sup>14</sup>

### 9.3.6.4 NEGATIVE RESULTS

---

If the chromatogram contains no peaks or the mass spectra of all peaks in the sample's chromatogram do not visually match any available reference material spectra the results shall be recorded in the case notes. (e.g., "no peaks", "no significant peaks", "no match", "no ID", "no controlled substances detected (NCSD)"). If the chemist desires to list the best library search result for components, the result must be enclosed in brackets.

## 9.4 GAS CHROMATOGRAPHY-RETENTION TIME (GCRT)

### 9.4.1 SCOPE

---

Gas Chromatography is used qualitatively for its ability to measure retention times of analytes which can be compared to retention times of known reference materials.

#### Instrument Operation Parameters

Instrument operation parameters are only one factor in obtaining a good separation. A wide variety of parameters may be adjusted by the chemist, with many combinations of parameters producing acceptable separation. The chemist should rely on their education and training concerning the theoretical and practical aspects of gas chromatography in the selection of instrumental parameters. A separation in the resulting chromatogram should be evaluated on the basis of efficiency (the narrowness of the peaks), the peak shapes (e.g., whether they tail or front) and the resolution represented.

Some of the acquisition conditions that the chemist may adjust are parameters such as: injection volumes, injector mode (e.g., split, splitless), temperature [e.g., of the inlet or oven (initial & final, ramps)], and flow rates. Regardless of the actual instrumental conditions the chemist uses, those conditions must be documented so that the resulting data could be reproduced if necessary.

Instrument parameters shall be in the case record either on the chromatographic data or included separately. If included separately from the chromatographic data, the method shall be identifiable to the sample for which it was used.

---

<sup>14</sup> Known breakdown products and manufacturing byproducts that are controlled are excluded from this requirement. Controlled precursors are excluded from this requirement unless they are the only controlled substance detected or the highest charge.

## 9.4.2 REAGENTS, STANDARDS, AND CONTROLS

---

Controls and standards used within this test method are described within their portion of the quality assurance/control portion of this test method.

## 9.4.3 SAMPLE PREPARATION AND ACQUISITION

---

The sample(s) and any necessary reference material(s) should be prepared at approximately the same concentration. The sample and reference material should not be acidic or basic or contain any solid material. One or more blank(s) shall be prepared to appropriately assess the chemical(s), solvent(s), and reusable extraction equipment (e.g., beakers, separatory funnels, mortar and pestle). One prepared blank shall be run immediately before the sample with the same acquisition parameters. Blanks are not required to be run before reference materials.

The reference material used for comparison can be run before or after the sample, as long as it has the same acquisition parameters.

When confirming  $\Delta 8$ -THC, if  $\Delta 8$ -THC and  $\Delta 9$ -THC are both found within a sample and there is concern of conversion, for the GCRT both reference materials shall be run to ensure there is no conversion from  $\Delta 9$ -THC to  $\Delta 8$ -THC occurring.

Reference materials must be run the same date of sample  $\pm 1$  day.

## 9.4.4 QUALITY ASSURANCE/CONTROL MEASURES

---

Because this test is performed on GCMS instrumentation, the quality assurance/control measures are the same as those listed in 9.3.4.

## 9.4.5 INTERPRETATION OF RESULTS

---

### 9.4.5.1 PRECAUTIONS TO BE TAKEN

---

#### INSTRUMENT:

- Performance check(s)
- Syringe or column blockage

#### SAMPLE PREPARATION:

- Poor choice of solvent (low analyte solubility) or extraction scheme
- Sample concentration is too dilute
- Insufficient sample taken for analysis
- The sample and reference material should not be acidic or basic or contain any solid material

#### RUNNING THE SAMPLE:

- Co-eluting compounds

- Improper acquisition parameters

#### 9.4.5.2 POSSIBLE SOURCES OF ERROR

- Co-eluting compounds
- Improper acquisition parameters
- Significant concentration difference between sample and reference material(s)
- Significant difference of peak start time between the sample and reference material(s)
- Heat induced conversion (e.g., NBOH compounds)

#### 9.4.5.3 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS

##### GENERAL

The chromatogram of the blank for samples must not contain any peaks (signal to noise  $\geq 5$ ) that have a positive retention time match to the reference material or sample's analyte of interest and also have an indicative mass spectrum for the analyte of interest. The analyte peak of the chromatograms for samples and reference materials must have a signal-to-noise ratio  $\geq 15$ .

The qualitative analysis of an unknown substance by GC is accomplished by matching the retention time of an unknown sample to the retention time of a known reference material using the following

calculation: 
$$\%_{RRT} = \left| \frac{t_{\text{sample}} - t_{\text{reference material}}}{t_{\text{reference material}}} \right| \times 100$$

The acceptable tolerances are listed in the table below (Table 9.3.5.3).

<b>TABLE 9.3.5.3 Maximum Retention Time Match Tolerances</b>	
<b>Retention Time</b>	<b>Tolerance</b>
$\leq 3$ minutes	2% relative
$> 3$ minutes	1% relative

##### 9.4.5.3.1 CRITERIA FOR POSITIVE RESULTS

The calculated relative retention time of the sample versus reference material is less than or equal to the acceptable tolerance listed in TABLE 9.3.5.3 and the analyte peak of the chromatograms for sample and reference material has a signal-to-noise ratio  $\geq 15$ .

##### 9.4.5.3.2 CRITERIA FOR NEGATIVE RESULTS

The calculated relative retention time of the sample versus reference material is greater than the acceptable tolerance listed in TABLE 9.3.5.3 and/or the analyte peak of the chromatogram for sample and/or reference material has a signal-to-noise ratio  $< 15$ .

## 9.4.6 REQUIRED DOCUMENTATION FOR GC QUALITATIVE RESULTS

---

### 9.4.6.1 GENERAL

For exhibits subjected to more than one GC test, the chemist will develop a way to relate the sample preparation/test results in the case notes to the corresponding instrumental printout(s).

The sample's peak of interest mass spectrum, used for retention time, shall be indicative (at minimum) and incorporated into the case record. If multiple reference materials are mixed in a vial, the mass spectrum for the comparison reference material, used for its retention time, shall be incorporated into the case record.

The relative retention time calculation(s) and reference material designation(s) shall be incorporated into case file. Calculations must be done on *DRG-FORM-30 RT Calculations*. If the retention time of the sample and reference are the exact same number, no calculation worksheet is necessary.

#### INSTRUMENTATION:

Images of chromatograms (blanks, reference materials & samples) supporting the analyst's conclusions must be incorporated into the electronic case file (e.g., by scanning or printing to the JusticeTrax Indexer program) before the case request status is marked 'Draft Complete' in JusticeTrax. The unique ASCL case number, exhibit number, date, and vial number must be visible on the image. The exhibit number is not required on reference material blanks, which are optional, and reference materials. For runs utilizing a nonstandard method, a copy of the instrumental parameters (method) must also be incorporated into the electronic case file (Only one copy per method per case file is necessary and must be treated as examination records).

### 9.4.6.2 POSITIVE RESULTS (GC QUALITATIVE)

Positive test results shall be recorded in the case notes in a manner similar to "Positive (+) retention time ( $t_R$ ) match for *compound*" or by listing the *compound*. If a compound is identified as positive and it has known closely eluting compounds, the start time report shall be incorporated into the case record. (e.g., o,m,p isomers, some nitazene compounds, etc.)

### 9.4.6.3 NEGATIVE RESULTS (GC QUALITATIVE)

Negative test results shall be recorded in the case notes in a manner similar to "Negative (-) retention time ( $t_R$ ) match for *compound*" or "No peak for comparison to analyte of interest".

## 9.5 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

### 9.5.1 SCOPE

The methods in this document describe various techniques used to prepare samples and obtain infrared spectra, precautions, and possible sources of error, data interpretation, and notations specific to this test. This test is intended to be supportive of other testing in most instances.

### 9.5.2 REAGENTS, STANDARDS, AND CONTROLS

Controls and standards used within this test method are described within their portion of the quality assurance/control portion of this test method.

### 9.5.3 SAMPLE PREPARATION AND ACQUISITION

#### ACQUISITION PARAMETERS

Routine Instrument Parameters for FTIR*	
Number of Scans	8
Resolution	4.000 cm <sup>-1</sup>
Sample Gain	Auto
Scanning Range	4000-400 cm <sup>-1</sup>
* These shall be considered starting point values only and may be adjusted by the chemist depending on the type of information needed.	

#### 9.5.3.1 ATTENUATED TOTAL REFLECTANCE (ATR) EXPERIMENTS

##### PREPARATION

Normally no sample preparation is needed to acquire infrared spectra of samples in ATR experiments. If an extraction is necessary to remove specific interfering substances, this will be documented in the case notes.

##### ACQUISITION

A blank spectrum shall be acquired before every sample, by screwing the anvil down onto the diamond crystal and acquiring a spectrum.

##### SOLID SAMPLES

Solids are applied directly to the diamond crystal. The anvil is screwed down into position forcing the sample against the crystal. The spectrum is acquired.

## LIQUID SAMPLES

Liquids are applied directly to the diamond crystal. Since liquids fully coat the crystal no pressure from the anvil is required. Volatile liquids may be covered with the supplied cover to prevent evaporation. The spectrum is acquired.

### 9.5.3.2 TRANSMISSION EXPERIMENTS – VAPOR PHASE TECHNIQUE

---

#### ACQUISITION & PREPARATION

A blank spectrum shall be acquired by placing a clean vapor phase cell in the sample chamber before each vapor phase IR sample (a vapor phase cell can be cleaned by wiping the cell with a clean wiping paper and heating the cell).

After the blank is acquired, the sample may be prepared in one of the following ways:

- A piece of wiping paper or a piece of filter paper is placed in the cell (in a manner that will not impede the IR beam) and a few drops of the sample solvent are placed on the paper.
- The vapor cell is held over the volatile liquid for a few seconds and the cell is closed.

Once the sample is prepared, the cell is placed in the sample chamber and the spectrum is acquired.

### 9.5.4 QUALITY ASSURANCE/CONTROL MEASURES

---

The maintenance and performance verification requirements are listed below for all FTIR instruments.

#### MAINTENANCE

Maintenance will be performed when the instrument's performance deems maintenance necessary and shall be recorded on *DRG-FORM-06*.

#### PERFORMANCE VERIFICATION

The performance verification of each FTIR must be checked each month the instrument is used and after any maintenance has been performed. There are many ways to verify that the instrument is functioning properly depending on the instrument model, instrument location (Little Rock or Lowell), and software version. The FTIR bench and ATR accessory are separate accessories and may require independent verification depending on need. If only the FTIR bench is in use, then verification of the bench is all that is needed. If the ATR accessory is in use, both the bench and ATR accessory must pass verification. Treat all ATR accessories gently when removing or inserting them into the FTIR.

Regardless of the instrument, the results of the performance checks shall be indexed into the instrument case record and recorded on *DRG-FORM-06*. If the instrument fails the performance check, additional checks may be performed. The instrument shall be removed from service if a

passing result cannot be obtained. This verification shall be performed prior to returning an instrument to service.

**Nicolet iS20 FTIR with ATR Accessory:**

1. Remove the ATR accessory.
2. Align the bench (>Collect> Experiment Setup>Diagnostic>Align).
3. Run the Valpro Qualification (>Analyze>Valpro Qualification> Nicolet iS20 KBr – Factory (CP, JP, PHEUR, PV, USP).
4. A ValPro Qualification Report will be generated. If all tests have passed, index the qualification report to the current year’s electronic case record for the instrument. Document the results of tests on the logsheet.
5. Replace the ATR accessory on the instrument.
6. Run the Valpro Qualification (Analyze>Valpro Qualification> Smart iTX accessory – PHEUR). When prompted, place the polystyrene standard on the diamond crystal and use the anvil to tighten down into position.
7. A ValPro Qualification Report will be generated. Index the qualification report to the current year’s electronic case record for the instrument. Document the results of test on the logsheet.
8. If the instrument failed any of these tests, the instrument must be removed from service for repair.

**Nicolet iS10 FTIR with ATR Accessory – Little Rock:**

1. Remove the ATR accessory.
2. Place the transmission plate in the FTIR (a screen should appear indicating ‘transmission experiment setup’)
3. Align the bench (>Collect> Experiment Setup>Diagnostic>Align).
4. Run the Valpro Qualification (>Analyze>Valpro Qualification> Nicolet iS10 KBr – EP).
5. A ValPro Qualification Report will be generated. If all tests have passed, index the qualification report to the current year’s electronic case record for the instrument. Document the results of tests on the logsheet.
6. Remove the transmission plate and replace the ATR accessory on the instrument.
7. Run the Valpro Qualification (Analyze>Valpro Qualification> Smart Diamond Accessory-EP). When prompted, place the polystyrene standard on the diamond crystal and use the anvil to tighten down into position.
8. A ValPro Qualification Report will be generated. Index the qualification report to the current year’s electronic case record for the instrument. Document the results of test on the logsheet.
9. If the instrument failed any of these tests, the instrument must be removed from service for repair.

## 9.5.5 INTERPRETATION OF RESULTS

---

### 9.5.5.1 PRECAUTIONS TO BE TAKEN

#### INSTRUMENT

- Verify that all necessary performance verifications have been done and that the instrument has passed each one

- Poor bench alignment, which is characterized by:
  - For a background spectrum, the %T at 4000  $\text{cm}^{-1}$  approaches zero
  - and/or after a sample spectrum has been baseline corrected, the baseline still “rolls” (e.g., the sample peaks appear on top of a decaying sinusoidal wave)

## SAMPLE PREPARATION

- Sample(s) are prepared in too dilute a form (**IDEAL:** The strongest peak will have an absorbance of at least 0.6 or %T of 25.)
- Sample(s) are prepared in too concentrated a form (**IDEAL:** The strongest peak will have an absorbance of no more than 1.2 or %T of 6.)
- Samples containing interfering compounds may require an extraction or other clean-up procedure to remove the interference

## RUNNING THE SAMPLE(S)

- Unusual matches suggested by the software matching algorithm: Check which search libraries are selected
- The spectrum contains incompletely subtracted background peaks (e.g.,  $\text{H}_2\text{O}$  absorptions at 3800 and 1600  $\text{cm}^{-1}$ , and  $\text{CO}_2$  absorptions at 2350 and 668  $\text{cm}^{-1}$ ): Collect a new background and re-run sample

### 9.5.5.2 SOURCES OF ERROR

---

- Interfering compound
- Insufficient subtraction of interfering compound
- Not enough sample for acquisition

### 9.5.5.3 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS

---

#### GENERAL

Identification of an unknown sample is based on comparing the sample's infrared spectrum with reference spectra. Care should be taken to evaluate the sample spectrum for peaks of interest prior to comparing to the reference spectrum<sup>15</sup>. Software matching algorithms are useful for rapidly narrowing the number of possible matches, but ultimate responsibility rests with the Forensic Chemist to determine whether a sample's infrared spectrum matches a given reference spectrum.

If a sample contains multiple infrared active compounds, extraction(s) or other clean-up techniques (or an entirely different testing technique) may need to be employed in order to positively identify these compounds.

Subtractions are permissible provided that the chemist includes the following data in the case file:

---

<sup>15</sup> The acceptable FTIR library for casework is the ASCL FTIR library. Other libraries may be used for investigative purposes and need an exception if used for casework reference matches.

- A printout of the original full sample spectrum with or without library match
- A printout of the full subtracted spectrum
- A printout of the subtraction results

An acceptable blank will not be a positive match for a controlled substance, a drug, or a common cutting agent, or contain any significant peaks.

#### 9.5.5.3.1 POSITIVE RESULTS

The sample's infrared spectrum visually matches that of the reference material spectrum. All peaks present in the reference material spectrum are also present in the sample's spectrum with exception of peaks in the sample that may be masked by interfering compounds.

#### 9.5.5.3.2 NEGATIVE RESULTS

The sample should be called negative if:

- The sample visually matches the reference material spectrum, but one or more reference material peaks are missing in the sample spectrum (This does not include peaks that could be masked by interfering compounds.).
- The sample doesn't visually match any available reference material spectrum

### 9.5.6 DOCUMENTATION REQUIREMENTS

---

#### 9.5.6.1 GENERAL

For exhibits subjected to more than one IR test, the chemist will develop a way to relate the sample preparation/test results in the case notes to the corresponding spectral image in the electronic case file.

#### CASE NOTES

The Forensic Chemist will include in the case notes or instrumental printout, for each IR test performed, the following information:

- Type of technique (ATR assumed unless otherwise noted)
- Type of sample preparation (Direct assumed unless otherwise noted)

#### INSTRUMENT PRINTOUTS

Images of spectra supporting the analyst's conclusions must be incorporated into the electronic case file (e.g., by scanning or printing to the JusticeTrax Indexer program) before the case request status is marked 'Draft Complete' in JusticeTrax. The unique ASCL case number, exhibit number, acquisition date, and acquisition parameters must be visible on the image.

### 9.5.6.2 POSITIVE RESULTS

---

Any compound(s) or mixture meeting the criteria for positive results will be entered into the case notes by chemical name or an appropriate abbreviation.

### 9.5.6.3 NEGATIVE RESULTS

---

Samples meeting the criteria for negative results will be entered into the case notes with a clear designation such as “No match” or “No ID.” If the chemist desires to list the best software algorithm match(s) of the sample spectrum to available library reference spectra, the match(s) shall be enclosed in brackets (e.g., [sodium bicarbonate]).

## 9.6 THIN LAYER CHROMATOGRAPHY

### 9.6.1 SCOPE

---

Thin layer chromatography is a separation technique used within Forensic Chemistry. The methods in this document describe the selection of a TLC solvent system, various aspects of the technique, precautions and possible sources of error, data interpretation and notations specific to this test. If compounds have the same retention factor, TLC may be used if the interfering compound is 2% or less of the peak area of the target compound on GCMS (e.g.,  $\Delta$ 8-THC and  $\Delta$ 9-THC, methamphetamine and MDMA, fluorofentanyl and fentanyl). The TLC retention factor of tianeptine methyl ester is different than that of tianeptine. Therefore, it is required that both the sample and RM are processed with the esterification procedure. Underivatized tianeptine has an insufficient retention factor for TLC to be an appropriate test.

### 9.6.2 REAGENTS, STANDARDS, AND CONTROLS

---

Specific reagents or controls are described elsewhere within this test method.

#### 9.6.2.1 SOLVENT SYSTEMS FOR TLC

---

A wide variety of solvent systems are described in TLC literature. The following table lists the most common solvent systems used; however the use of any published TLC solvent system is acceptable.

<b>Common TLC Solvent Systems</b>		
<b>System</b>	<b>Makeup</b>	<b>Useful For...</b>
Davidow	Davidow solution <sup>1</sup> : Ammonium Hydroxide (95:5)	Wide variety of acidic, basic and neutral drugs
T1	Methanol: Ammonium Hydroxide (95:5)	Wide variety of acidic, basic and neutral drugs
Hexane/Ether	Hexanes <sup>2</sup> : Diethyl Ether (80:20)	Cannabinoids
Pet Ether/Ether	Petroleum Ether: Diethyl Ether (85:15)	Cannabinoids – better separation of Δ8-THC and Δ9-THC
Steroids	Methylene Chloride <sup>3</sup> : Ethyl Acetate (80:20) <b>OR</b> Methylene Chloride <sup>3</sup> : Methanol (90:10)	Steroids
<sup>1</sup> Ethyl Acetate: Methanol (85:10). <sup>2</sup> Petroleum Ether or Ligroin may be substituted. <sup>3</sup> Chloroform may be substituted.		

Usually a 100 mL portion of the selected solvent system is prepared and transferred to a labeled glass tank lined with filter paper and fitted with a lid.

### 9.6.3 SAMPLE PREPARATION AND ACQUISITION

#### 9.6.3.1 SAMPLE PREPARATION

Solid samples are dissolved in an appropriate solvent (samples being subjected to Hexane/Ether thin layer shall be extracted in methanol unless methanol is inappropriate). Liquid samples may be used as is or diluted in an appropriate solvent. Some samples may require an extraction procedure to remove interfering compounds.

#### 9.6.3.2 SAMPLE AND REFERENCE MATERIAL APPLICATION

- A line is drawn with a pencil parallel to, and at least 2 cm from, the bottom of the TLC plate
- The samples are spotted on this line, called the origin, starting at least 2 cm from the side of the plate and at least 1 cm from each other
- The sample and reference material spots are labeled uniquely. The RM must be labelled with its entire designation. The chemist must be able to correlate the sample spot with the case and exhibit number.
- The sample(s) and reference material(s), in an adequate volume, are applied with separate capillary tubes
- CBD reference material shall be run on the plate if GCMS results are positive for CBD and Δ9-THC in the sample and no semi-quant analysis has been performed
- Thin layer plates must be labelled with analyst's initials and date it is run in the tank

#### 9.6.3.3 RUNNING THE PLATES

- The TLC plate is placed in a vertical position in a tank containing the selected solvent system so that the application line (origin) is above the level of the mobile phase

- Prepared tanks shall be refreshed before use every three hours at minimum.
- Normally the plate is allowed to develop through a distance of 10-15 cm
- When the development period is complete, the plate is removed from the tank and allowed to dry before visualization

#### 9.6.3.4 VISUALIZATION FOR TLC

As most organic compounds are colorless, they must be made visible so that their retention factors can be compared, preferably by a non-destructive technique. There are a wide variety of visualization techniques available, depending on the compound of interest. Visualization techniques that are generally used are described in the table below.

TLC plates run for the analysis of items suspected to contain THC shall

1. Contain the appropriate cannabinoid reference materials
2. Have their baselines covered during the application of Fast Blue BB spray
3. Have the baseline sprayed with acidified iodoplatinate.

If the visualization of an item with acidified iodoplatinate indicates (by comparison to the reference material usually on or near the baseline) that another controlled substance may be present, further testing (GCMS analysis at a minimum) is required.

<i>Common TLC Plate Developing Techniques</i>		
<b>Visualization Technique</b>	<b>Useful For...</b>	<b>Comments</b>
Ultraviolet (UV) Light	Wide variety of organic molecules	Use before any reagent indicator sprays, circle spots in pencil (NOTE: The ability to see a given compound may be pH dependent.)
Fast Blue BB	Cannabinoids	Heat plate after spraying
Ninhydrin	Primary and secondary amines	Heat plate after spraying
Acidified Iodoplatinate	Primary through tertiary amines, quaternary ammonium compounds	Useful for overspraying a plate previously sprayed with Ninhydrin or Fast Blue BB (cool plate before spraying)
PMBA	Ergot alkaloids, tryptamines	Heat plate after spraying (required for samples containing LSD and psilocin/psilocybin)
Ethanol: H <sub>2</sub> SO <sub>4</sub> (4:1)	Steroids	Heat plate after spraying

#### 9.6.3.5 PHOTOGRAPHY OF THIN LAYER PLATES

Thin layer plates shall be photographed to be incorporated into the case record to provide reviewable data. Each workstation has an SD card and an SD card reader. Photos shall be transferred to a folder on the network for safe keeping until corresponding cases are

administratively reviewed, at minimum. It is up to the analyst to decide how they intend to organize files on their SD card and network folder.

## 9.6.4 INTERPRETATION OF THE RESULTS

---

### 9.6.4.1 PRECAUTIONS TO BE TAKEN

#### SAMPLE PREPARATION

- Choose an appropriate solvent for the analyte(s) of interest
- Perform extractions on samples that may contain interfering compounds

#### SAMPLE APPLICATION

- Spot shall be no more than ~4 mm in diameter or resolution will be lost
- The plate surface should not be cut or gouged by the applicator
- It is essential that the spot be dry at the end of application, especially if the solution contains water. Even a small amount of a polar solvent adsorbed on the plate can drastically alter chromatographic properties.

#### RUNNING THE PLATES

- Overdeveloping the plates may lead to excessive zone (spot) broadening causing secondary problems such as:
  - Weak samples may “disappear”
  - Concentrated samples may overlap with spots in neighboring lanes
- Under developing the plate will result in poor separation for complex samples
- Use of stale solvent system tanks or the improper selection of solvent system may result in poor chromatography

#### VISUALIZATION

The maximum amount of data is gained from a TLC plate when multiple visualization techniques are used. Poor planning on the order of visualization techniques may lead to data loss.

### 9.6.4.2 SOURCES OF ERROR

#### SAMPLE PREPARATION

- Sample is too dilute resulting in no spot
- Poor choice of solvent for analyte resulting in no spot

#### SAMPLE APPLICATION

- Failure to spot the appropriate or any reference material on the plate

## VISUALIZATION

- Sample contains two compounds with similar retention factors and they overlap upon visualization (examples listed below):
  - meth/MDMA/codeine
  - $\Delta$ 8-THC/ $\Delta$ 9-THC/(6aR,9S)  $\Delta$ 10-THC (H/E tank)
  - (6aR,9R)  $\Delta$ 10-THC / $\Delta$ 6a,10a-THC (H/E tank)
  - $\Delta$ 8-THC/ $\Delta$ 8-THC acetate/ $\Delta$ 9-THC C8/ $\Delta$ 8 iso-THC/ $\Delta$ 9(R)-hexahydrocannabinol/THCP/THCH (Pet E/E tank)
  - $\Delta$ 9-THC/ $\Delta$ 9-THC acetate/THCB (THCP/THCH depending on concentration) (Pet E/E tank)
  - cocaine/fentanyl/multiple fentanyl analogs/eutylone
  - 4-acetoxy DMT and psilocin
- Reference materials do not produce visual spots
- Waited too long to spray plate with visualization reagent

### 9.6.4.3 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS

---

#### 9.6.4.3.1 GENERAL

---

Identification of compounds by TLC is accomplished by matching the retention factors and visualization reaction(s) of known reference materials run simultaneously and on the same plate as the samples.

If a sample contains compounds with similar retention factors and visualization reactions, selection of a different solvent system and/or visualization techniques (or an entirely different testing technique) may need to be employed in order to differentiate these compounds.

#### 9.6.4.3.2 CRITERIA FOR POSITIVE RESULTS

---

A compound in a sample matches the retention factor and visualization reaction(s) of a reference material on the same plate.

#### 9.6.4.3.3 CRITERIA FOR NEGATIVE RESULTS

---

No spots that match a reference material are visible in the sample lane or no spots are present in the sample lane.

## 9.6.5 DOCUMENTATION REQUIREMENTS

---

### 9.6.5.1 GENERAL

---

The Forensic Chemist will include in the case notes, for each TLC test performed, the following information:

- Type of solvent system used

- Visualization technique(s) employed (e.g., UV, Ninhydrin)
- The case record shall also contain a photograph for each TLC test performed. The photo shall allow for the labelling, date, and resulting spots to be viewed by anyone reviewing the case record.
  - The date of spraying is considered to be the same as the date on the TLC plate unless otherwise noted in the notes

#### 9.6.5.2 DOCUMENTATION OF POSITIVE RESULTS

---

Any compound(s) meeting the criteria for positive results will be entered into the case notes by chemical name or an appropriate abbreviation.

#### 9.6.5.3 DOCUMENTATION OF NEGATIVE RESULTS

---

Any sample(s) meeting the criteria for negative results will be entered into the case notes in one of the following ways. Samples containing no spots may be entered as “negative” or “no spots”. If spots are present, the number of spots shall be recorded in the notes.

### 9.7 PHARMACEUTICAL IDENTIFICATION

#### 9.7.1 SCOPE

---

The methods described in this document can be used to aid in the identification of pharmaceuticals, in the form of tablets and capsules, submitted for drug analysis. This method is used to presumptively identify commercial pharmaceutical products based off of characteristics/appearances only.

Instances in which Pharmaceutical Identification should be performed:

- The tablets are suspected to contain codeine, on any other multi-scheduled drug, where an ID can help determine which schedule to report
- Analyst suspects the tablets may be counterfeit but a one-to-one comparison is needed to make that conclusion
- Items being tested may all yield non-controlled results

#### 9.7.2 REAGENTS, STANDARDS, AND CONTROLS

---

There are no reagents, standards, or controls associated with this test.

### 9.7.3 SAMPLE PREPARATION

---

There is no sample preparation associated with this test. The physical appearance of the tablet/capsule (e.g., imprint, color, shape, scoring) is required to do the comparison and is required to be in the case notes.

### 9.7.4 QUALITY ASSURANCE/CONTROL MEASURES

---

The Forensic Chemistry section has a set list of reference sources that are allowed for pharmaceutical identification. Sources with a photo for comparison are the preferred method after the ASCL Retained Tablet Library. Acceptable sources are listed below:

- Arkansas State Crime Lab Retained Tablet Library (this is the preferred method of identification if a retained tablet is available)
- DIB (any year)
- IdentiaDrug (any year)
- Poison Control
- Drugs.com (pill identifier only)
- Manufacturer sources of information (packaging - United States products only, conversations, e-mails, and manufacturer produced ID books)

### 9.7.5 INTERPRETATION OF RESULTS

---

#### 9.7.5.1 PRECAUTIONS TO BE TAKEN/SOURCES OF ERROR

---

- Pharmaceuticals containing similar imprint information
- Counterfeit items
- Imprints matching multiple identifications
- No photo for comparison

#### 9.7.5.2 CRITERIA FOR POSITIVE, INDICATIVE, AND NEGATIVE RESULTS

---

##### 9.7.5.2.1 POSITIVE RESULTS

---

The active ingredient(s) of the tablet/capsule have been identified by matching the physical appearance of the tablet/capsule to a reference source.

##### 9.7.5.2.2 INDICATIVE RESULTS

---

Broken, partial, or worn tablets or damaged capsules largely match a reference source but some markings are not visible.

#### **9.7.5.2.3 NEGATIVE RESULTS**

---

The active ingredient(s) of the tablet/capsule could not be identified because information from reference sources failed to match the physical appearance of the tablet.

### **9.7.6 DOCUMENTATION REQUIREMENTS**

---

The imprint as well as color, shape, and/or scoring of the tablet/capsule must be documented in the case notes.

#### **9.7.6.1 POSITIVE RESULTS**

---

The active ingredient(s) of any tablet/capsule identifications meeting the criteria for positive results will be entered into the case notes by name or an appropriate abbreviation. The dosage and reference source(s) used to make the identification, and date of identification will also be documented in the case notes. For online sources, only the identification and date need to be recorded in the case notes. The online identification shall be indexed into the case record; the indexed file shall be identifiable to the compared item(s) and contain the date the identification was performed.

#### **9.7.6.2 INDICATIVE RESULTS**

---

The possible active ingredient(s) of any tablet/capsule identifications meeting the criteria for indicative results will be entered into the case notes by name or an appropriate abbreviation followed by a question mark, and the notation will be enclosed in parentheses. The dosage and reference source(s) used to make the identification, and date of identification will also be documented in the case notes. For online sources, only the identification and date need to be recorded in the case notes. The online identification shall be indexed into the case record; the indexed file shall be identifiable to the compared item(s) and contain the date the identification was performed.

#### **9.7.6.3 NEGATIVE RESULTS**

---

If a comparison is used to rule out an ID, the results, source, and date must be documented in the case notes. Notes shall also detail the reasons for reaching the negative conclusion. Results will be entered as “negative” or by putting the drug name in brackets. For online sources, only the result and date need to be recorded in the case notes. The comparison information shall be indexed into the case record; the indexed file shall be identifiable to the compared item(s) and contain the date the comparison was performed.

## 9.8 COLOR TESTING

### 9.8.1 SCOPE

---

The methods in this document describe different color tests commonly used and how to perform those color tests. The results of color tests are indicative of the presence or absence of various drug classes and/or organic functional groups. The particular color test(s) used by the Forensic Chemist are usually indicated by the type of sample. Color tests are normally used to help plan future testing of the sample (e.g., appropriate extraction method, instrumentation parameters). If color tests are performed and no additional testing is done, it must be clearly communicated on the report.

### 9.8.2 REAGENTS, STANDARDS, AND CONTROLS

---

There are multiple reagents used for color testing. They are listed within this method and the reference materials used to performance check them are listed within the reagent preparation instructions in Qualtrax. Controls, if used, are listed with the specific color test technique.

### 9.8.3 SAMPLE PREPARATION AND ACQUISITION

---

#### 9.8.3.1 GENERAL PROCEDURE

---

- ❶ Place the appropriate reagent(s) in a well plate depression (or a new test tube)
- ❷ Add a small amount of sample
- ❸ Add any additional reagents necessary (multiple reagent color tests)
- ❹ Examine the reactants for any changes
- ❺ Record observations in the case notes

#### 9.8.3.2 COMMON COLOR TESTS

---

Color tests routinely used are listed below along with any specific modifications to the above method. The analyst is not limited to the following list of color tests. Other published and recognized color test(s) are acceptable and may be used as needed.

##### 9.8.3.2.1 SINGLE REAGENT COLOR TESTS

---

- Marquis
- *p*-dimethylaminobenzaldehyde (PMBA)

##### 9.8.3.2.2 MULTIPLE REAGENT COLOR TESTS

---

- Cobalt Thiocyanate/Stannous Chloride  
In step ❶ add Cobalt Thiocyanate, add Stannous Chloride in step ❸

#### 9.8.3.2.3 MODIFIED DUQUENOIS-LEVINE

This test is appropriate for testing for cannabinoids. Solvents normally used for the extraction include pet ether, hexanes, ligroin, methanol, etc.

1. Transfer a portion of the extract to a new labeled test tube. Heat the test tube to reduce the solvent volume if necessary
2. Add approximately 1 mL of Duquenois-Levine reagent
3. Add approximately 1 mL of concentrated hydrochloric acid (HCl)
4. Agitate the solution and observe any color change
5. Add approximately 1 mL of methylene chloride to the solution and agitate
6. Observe the color of the bottom layer and record in the case notes
7. Run  $\Delta^9$ -THC positive control along with samples. Follow steps 1-5. Record  $\Delta^9$ -THC reference material designation used in notes

#### 9.8.3.2.4 NESSLER'S - TESTING FOR AMMONIA GAS

1. Draw a portion of Nessler's supernatant into pipet
2. Draw a portion of sample vapor into pipet, while making sure not to make contact between the pipet or its contents and the sample
3. Observe the color change in the pipet
4. Record in the case notes

#### 9.8.3.2.5 FLAME TEST

When a metal salt is introduced into the flame of a Bunsen burner, the metallic ion produces characteristic color in the flame.

1. In a safe area, ignite a Bunsen burner
2. Obtain a piece of nichrome wire with a loop in one end
3. To clean the wire first dip the wire loop into 0.1N hydrochloric acid and then into deionized water
4. Heat the wire loop in the Bunsen burner flame until the wire begins to glow. Repeat steps 3 & 4 until no color is observed in the flame
5. Dip the looped end of the wire into a sample. (The sample may be solid or dissolved in a small amount of deionized water. If the sample is to be used in a solid form it may be helpful to dampen the wire loop with dilute hydrochloric acid before dipping it in the sample.)
6. Place the loop at the tip of the inner cone of the flame and observe the color given off and record in the case notes

#### 9.8.3.2.6 PH TEST

This test is used primarily in manufacturing cases to determine whether a liquid is acidic or basic.

#### PROCEDURE:

1. Test litmus paper with acid/base to ensure the paper is still working as expected
2. Deposit a small amount of the solution to be tested onto the litmus paper
3. Compare the color on the litmus paper with the pH scale provided on the litmus paper packaging

4. Record the results in the case notes

#### 9.8.3.2.7 4-AMINOPHENOL COLOR TEST FOR CANNABIS PLANT MATERIAL

This is a presumptive screening test for plant material that may aid in efficient sampling of plant material cases. A color reaction of pink or blue is observed depending on the concentration of certain cannabinoids. A pink color<sup>16</sup> can indicate cannabidiol (CBD) is in higher concentration than other cannabinoids, while a blue color can indicate  $\Delta$ 9-tetrahydrocannabinol ( $\Delta$ 9-THC) is in higher concentration than other cannabinoids.

#### PROCEDURE:

1. Add approximately 25 mg of the plant material sample to a test tube
2. Add approximately 1 mL of methanol
3. Vortex for approximately 3 seconds
4. Add 1-3 drops of the extracted material to a new test tube
5. Add approximately 1 mL of 4-AP Solution A and 4 drops of 4-AP Solution B to the test tube
6. Vortex for approximately 3 seconds
7. Document color result after 2 minutes (if the color is not discernibly pink or blue, a result of inconclusive is acceptable)

If only this color test is performed, there is no need to record a reserve weight as we know approximately how much is necessary for the test. It is also assumed that a methanol extraction was performed, therefore, it is not required to be recorded in the notes unless it will also be used for other testing.

Sources of error specific to this color test include but are not limited to:

- Too much or not enough plant material used for the test can affect the color intensity
- Too much methanol added for extraction can affect the color intensity
- Too much 4-AP Solution A can affect the color intensity
- Recording results before sufficient time has elapsed (up to 5 minutes was tested in the validation and found to be consistent with the suggested 2 minutes)

Known limitations:

- Blue color is observed when CBN,  $\Delta$ 8-THC, THCV, or  $\Delta$ 9-THC is the most abundant cannabinoid
- Pink color is observed when CBD or CBG is the most abundant cannabinoid

These limitations should have no bearing on the overall outcome of the testing, since this color test is used as a guide on sampling and further testing.

---

<sup>16</sup> The color result of the test does not necessarily correlate with whether the sample is hemp or marihuana, as some samples were found to have a higher CBD concentration and yielded a pink color, but were determined to be marihuana after full testing during the validation.

## 9.8.4 QUALITY ASSURANCE/CONTROL MEASURES

---

All reagents are verified prior to use in case work. The Duquenois-Levine test has a positive control run with it at the time of the test. The application of the reagents to the spot well or test tube acts as a negative control or blank.

## 9.8.5 INTERPRETATION OF RESULTS

---

### 9.8.5.1 PRECAUTIONS TO BE TAKEN

---

- Ensure correct order of addition of reagents, if it is a multiple reagent test
- Ensure sufficient sample is introduced to the spot well for testing
- Make sure reagent is added before sample

### 9.8.5.2 SOURCES OF ERROR

---

- Dirty spot well could result in false positive
- Poor choice of color test for sample
- Reagent is close to or beyond its usable timeframe
- Color of sample or liquid may interfere with observed results

### 9.8.5.3 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS

---

A color change is considered a positive result. No color change is considered a negative result. For pH test, there is no positive or negative result; the analyst will record the pH as the number on the scale the observed color most closely matches. For the 4-aminophenol color test, a result of inconclusive may also be acceptable if differentiation between pink and blue cannot be made.

## 9.8.6 DOCUMENTATION REQUIREMENTS

---

The case notes shall include:

- The test performed
- The date of the test
- The result of the test

## 9.9 MORPHOLOGICAL MICROSCOPY OF PLANT MATERIAL

### 9.9.1 SCOPE

---

This method is employed to examine plant material for the presence of cystolithic hairs.

## 9.9.2 REAGENTS, STANDARDS, AND CONTROLS

---

N/A

## 9.9.3 SAMPLE PREPARATION

---

Direct microscopic examination is the most routine type of analysis. A solvent rinse may be employed, if the sample is moldy, degraded, burned/covered in ash, or very resinous.

## 9.9.4 QUALITY ASSURANCE/CONTROL

---

Microscopes are cleaned and/or serviced as needed.

## 9.9.5 INTERPRETATION OF THE RESULTS

---

The presence of the following features may be noted:

- Longitudinally grooved stalks and stems
- The top surface (adaxial) of the leaves are darker than the bottom surface (abaxial)
- Compound, palmate leaves with an odd number of leaflets (typically seven)
- Leaflets are serrated (pointing toward tips) and pointed at both ends
- Ovoid, mottled seeds (typically brown) that have a ridge around the greatest circumference
- Seeds contain a white flesh that resembles coconut flesh
- Glandular trichomes (with or without stalk, bulbous)
- Non-glandular trichomes (non-cystolithic)

### 9.9.5.1 PRECAUTIONS TO BE TAKEN AND POSSIBLE SOURCES OF ERROR

---

- Not acquiring a sufficient sample for examination (e.g., size of sample or quality of sample taken)
- Not prepping a sample that needs rinsing
- Dirty microscope oculars
- Inadequate lighting
- Improper focus
- Sample is too finely ground

### 9.9.5.2 CRITERIA FOR POSITIVE AND NEGATIVE RESULTS AND REQUIRED DOCUMENTATION

---

#### 9.9.5.2.1 POSITIVE RESULTS

---

Presence of cystolithic hairs attached to leaf material is a positive result for microscopic examination. The results shall be recorded in the case notes.

#### 9.9.5.2.2 NEGATIVE RESULTS

Absence of cystolithic hairs attached to leaf material is a negative result for microscopic examination. The results shall be recorded in the case notes.

### 9.10 SEMI-QUANTITATIVE DETERMINATION OF $\Delta$ 9-THC

#### 9.10.1 SCOPE

This method is for semi-quantitative evaluation of  $\Delta$ 9-tetrahydrocannabinol ( $\Delta$ 9-THC) in plant material, using liquid extraction and agitation followed by analysis on a gas chromatograph-mass spectrometer (GCMS). A qualitative result may be determined by comparison to the response of a decision point standard, using the internal standard tribenzylamine (TBA).

This method should only be employed where the individual item(s) to be tested contains at least 0.5g of loose plant material.

#### 9.10.2 REAGENTS, STANDARDS, AND CONTROLS

Certified reference materials shall be used to establish traceability. These will be purchased from a provider that is ISO 17034 accredited.

##### Decision Point Preparation

1. Pipet 500  $\mu$ L of  $\Delta$ 9-THC CRM into an auto-sampler vial using a calibrated micropipette
2. Add 1000  $\mu$ L of TBA semi-quant internal standard solution using a calibrated micropipette
3. Cap vial and agitate to mix

##### Extracted Blank Preparation

1. Add 1000  $\mu$ L of semi-quant TBA internal standard solution to an auto-sampler vial
2. Cap vial

#### 9.10.2.1 SAMPLE AND POSITIVE CONTROL PREPARATION

1. Weigh out 95–105 mg of plant material<sup>17</sup> and add it to a test tube
2. Record the amount in the case record
3. Add 2000 $\mu$ L of the TBA semi-quant internal standard solution using a calibrated micropipette
4. Vortex for approximately one minute and allow to extract for ten minutes
5. Filter extract and transfer approximately 1 mL to an auto-sampler vial and cap

<sup>17</sup> For the purpose of semi-quant, plant material is defined as leaf material only and does not include stems or seeds.

Sample preparation and acquisition may be carried out by another competent analyst to facilitate batching samples for analysis. Transfer of the pre-weighed sample to the analyst performing the extraction shall be noted on the Semi-Quant batch worksheet.

#### 9.10.2.2 ACQUISITION

---

The data is acquired using the THC method in scan mode.

A cannabidiol (CBD) RM shall bracket each batch sequence to determine if conversion to  $\Delta$ 9-THC has occurred. A passing CBD RM shall have no indicative or positive  $\Delta$ 9-THC peaks. If  $\Delta$ 9-THC from conversion meets the criteria for positive or indicative, samples that have higher CBD abundance than  $\Delta$ 9-THC must be rerun with proper controls.

A positive control (PC) shall bracket each batch sequence to evaluate if the extraction procedure was performed properly and if the instrument is performing properly. A passing PC shall have an acceptable front PC blank, response of +/- 0.1 of known  $\Delta$ 9-THC concentration, and front/back  $\Delta$ 9-THC responses within 0.03 of each other. It is allowable for the back PC blank to be unacceptable as long as the front/back PC  $\Delta$ 9-THC calculations to the known and each other pass.

CBD and PC shall be run on the same liner.

An extracted blank (negative control) will be run with each batch, to evaluate the materials used in the extraction for contamination. This sample comprises only the materials used in the extraction, with no plant material added. An acceptable extracted blank will not contain any peak in the retention time area of  $\Delta$ 9-THC with indicative mass spectrum for  $\Delta$ 9-THC or any other peak (signal to noise  $\geq$  5) whose mass spectrum is a positive or indicative match for a controlled substance, drug, or common cutting agent. A solvent blank shall be run before each case sample to detect carryover. An acceptable blank will not contain any peaks (signal to noise  $\geq$  5) whose mass spectrum is a positive or indicative match for a controlled substance, drug, or common cutting agent.

A solvent clean shall be run after each extracted case sample to help prevent carryover.

A decision point shall be prepared, using a  $\Delta$ 9-THC CRM and the TBA stock solution, and run before each batch of samples.

The sequence format for a decision point and samples is:

<b>Sequence order</b>	<b>Sample</b>	<b>Purpose</b>
1	CBD RM	Liner conditioner
2	CBD RM	Evaluate for Δ9-THC conversion
3	Solvent clean	Clean inlet/column
4	Extracted blank	Evaluate extraction process for contamination
5	Solvent clean	Clean inlet/column
6	Solvent blank	Evaluate for carryover
7	Decision Point	Establish Decision Point
8	Solvent clean	Clean inlet/column
9	Solvent blank	Evaluate for carryover
10	Positive Control	Evaluate for extraction effectiveness and instrument function
11	Solvent clean	Clean inlet/column
12	Solvent blank	Evaluate for carryover
13	Case 1	Analyze sample
14	Solvent clean	Clean inlet/column
15	Solvent blank	Evaluate for carryover
16	Case 2	Analyze sample
17	Solvent clean	Clean inlet/column
18	Solvent blank	Evaluate for carryover
19	Positive Control	Evaluate for extraction effectiveness and instrument function
20	Solvent clean	Clean inlet/column
21	CBD RM	Evaluate for Δ9-THC conversion

The decision point and extracted samples may be analyzed up to three days after their extraction/initial analysis. Extracted blank, positive control and CBD reference material data shall be stored in the appropriate JusticeTrax case file and their evaluation recorded on the batch worksheet via the verifier's initials.

### **9.10.3 QUALITY ASSURANCE/CONTROL MEASURES**

All QA/QC requirements for the GCMS instrument also apply for semi-quantitative testing with the exception of daily test mix. There are other controls in place to check this process.

#### **PIPETTE CHECKS**

Pipettes shall be checked by the analyst performing the test prior to use. A mid-range and high value shall be checked on the 100-1000 µL pipette used.

Pipette check procedure:

1. Obtain beaker of E-pure water
2. Take temperature of water
3. Record temperature on the appropriate pipette check spreadsheet
4. Enter the Z factor for recorded temperature into the appropriate cell on the spreadsheet (make sure you have it recorded as 1/Z factor)
5. Tare a beaker on the balance and begin to aliquot portions of liquid into the beaker
6. Record the weight on the spreadsheet of each portion for 10 repetitions

7. Check to make sure the % error is below the acceptable error in section 6.4.7.1 of this manual
8. Check to ensure the %CV is < 3.0%.
9. If the error or %CV is too high, the cause shall be evaluated and the check may be performed again
  - a. If it still fails, the pipette shall be taken out of service until maintenance is performed and two performance checks pass for each range
  - b. If it passes, perform a third check which must also pass. If it does not, the pipette shall be taken out of service until maintenance is performed and two performance checks pass for each range

## 9.10.4 INTERPRETATION OF RESULTS

---

### 9.10.4.1 PRECAUTIONS TO BE TAKEN

---

- Ensure complete integration of all analyte and internal standard peaks

### 9.10.4.2 POSSIBLE SOURCES OF ERROR

---

Potential errors in decision point preparation:

- Improperly pipetting reference material or internal standard
- Reference material concentration (e.g., bad batch/concentration is incorrect)

Potential errors in sample preparation:

- Incomplete transfer of sample to extraction vessel
- Improperly pipetting internal standard
- Inadequate extraction time/process

### 9.10.4.3 DATA INTERPRETATION

---

There must be no reportable analyte peak in the extracted blank (negative control) for the batch results to be acceptable.

An acceptable blank will not contain any peaks (signal to noise  $\geq 5$ ) whose mass spectrum is a positive or indicative match for a controlled substance, drug, or common cutting agent.

The data analysis method employed in ChemStation to capture the responses of  $\Delta 9$ -THC and TBA uses the following ions:

#### **$\Delta 9$ -THC**

- quantitative ion 299
- qualitative ions 231 and 314

## TBA

- quantitative ion 210
- qualitative ions 196 and 287

Semi-quant calculation shall not be run on any samples that do not have a positive mass spectrum for  $\Delta^9$ -THC.

### 9.10.5 REQUIRED DOCUMENTATION

---

Each case record shall contain:

- Decision point blank and sample data
- Casework blank and sample data
- Casework semi-quant report(s)
- Semi-quant results
- Batch worksheet with:
  - Preparation date
  - Case number and item number
  - Pipette used
  - CRM manufacturer and lot number
  - TBA designation
  - Initials and date documenting transfer of sample for analysis, if applicable
  - Verifier's initials for EB, PC, and CBD

## 9.11 EXTRACTIONS FOR DIFFICULT SAMPLES

### 9.11.1 REQUIRED EXTRACTIONS

---

#### **Illicit Tablets (suspected Ecstasy tablets)**

1. Sample approximately half of the tablet and homogenize it
2. Split into two test tubes
3. Extract the sample in enough solvent to fill a GCMS vial (base extraction is preferable and the analyst may use hexanes as a solvent to remove excess caffeine)
4. Filter into GCMS vial

**\*\*Do not concentrate without consultation with a section chief or technical leader\*\***

#### **Suspected NBOH Compounds Derivatization**

This extraction is necessitated because the NBOH compounds break down on GCMS to their respective 2-C counterparts. (e.g., 25C-NBOH will break down to 2C-C on GCMS) Thin layer

chromatography of the 2C compounds is significantly different from the NBOH counterpart compounds and will reveal the need to perform derivatization.

1. Dissolve the sample in Acetonitrile that has been dried over Sodium Sulfate.
2. Add dried Acetonitrile to an empty test tube (for a blank).
3. Remove the liquid from the sample extract and place in a labelled test tube. Add Sodium Sulfate to further dry sample.
4. Add ~160 µL of sample to a GCMS vial with sleeve.
5. Add ~160 µL of dried acetonitrile to a GCMS vial with sleeve.
6. Add ~160 µL of BSTFA (with 1% TMCS) to both vials.
7. Cap and mix well
8. Run on broad method
9. If you get poor results, repeat the process trying to reduce air/moisture.

#### **Items containing both 4-acetoxy DMT and psilocin**

It is known that 4-acetoxy DMT hydrolyzes to psilocin with some extraction types. If 4-acetoxy DMT and psilocin are found together in the same GCMS data, the below steps shall be followed.

1. Extract a new sample using acetonitrile
2. Run on GCMS and assess (if psilocin persists, consult a section chief or technical leader).

#### **Items suspected to contain LSD**

Acetonitrile is suggested as the main solvent for suspected LSD samples because it does not promote conversion of other LSD compounds to LSD (e.g., 1p-LSD)

1. Extract sample using acetonitrile
2. If no controlled substances are detected, dry down sample and reconstitute in methanol

#### **Items suspected to contain Tianeptine**

Shall be concentrated in dichloromethane and sleeved for GCMS analysis

### **9.11.2 SUGGESTED EXTRACTIONS FOR DIFFICULT SAMPLES**

---

#### **Clonazepam tablets**

1. Add saturated sodium bicarbonate
2. Extract with minimal methylene chloride and let soak for 1-2 hours

#### **LSD on sweet tarts or sugar cubes**

1. Soak in 0.1N HCl overnight
2. Make basic with saturated sodium bicarbonate
3. Extract into methylene chloride

4. Dry down in a sleeve

### **Mushroom Extraction**

1. Soak in 0.1 N HCl (for at least 4 hours)
2. Wash with methylene chloride
3. Take the 0.1 N HCl layer and add saturated sodium bicarbonate to make basic (pH ~ 9 or 10)
4. Add methylene chloride
5. Dry methylene chloride layer down to a sleeve

### **Mushroom/Chocolate Extraction**

1. Sample is transferred into a mortar and ground with a pestle.
2. The resulting powder is covered with 10 % acetic acid (or 0.1N HCl), and the sample is further ground with a pestle.
3. An additional 5 to 7 ml of distilled or E-Pure water is added, and the mixture is ground for about 2 minutes, creating a thin slurry.
4. This slurry is divided into equal portions and is transferred into large screw top tubes.
5. Approx. 3 ml of CH<sub>2</sub>Cl<sub>2</sub> is added to each tube, vortexed, and the tubes are centrifuged for 3 minutes.
6. The aqueous layer is pipetted into a beaker from each of the tubes.
7. The aqueous solution in the beaker is neutralized by slowly adding solid sodium bicarbonate until the effervescence stops.
8. The pH is checked with pH paper to make sure it is at least 9 and doesn't surpass it.
9. The resulting solution is then transferred into large screw top tubes, and each extracted with approx. 3 ml of CH<sub>2</sub>Cl<sub>2</sub>.
10. The tubes are vortexed then centrifuged for about 5 minutes.
11. The CH<sub>2</sub>Cl<sub>2</sub> layers are combined in a small beaker.
12. The CH<sub>2</sub>Cl<sub>2</sub> extract is concentrated under air (no heat), transferred to a sleeve, and analyzed on the GCMS.

### **THC Candy**

1. Dissolve sample in hot distilled or E-Pure water
2. Extract in Petroleum Ether.
3. Dry down.
4. Reconstitute in methanol.

### **THC Edibles**

Extract with acetonitrile.

### **Esterification Derivatization (for Tianeptine)**

1. Add 5 drops of concentrated HCl to about 0.5mL of methanol (MeOH) in a test tube
2. Add approximately 2.5 mg of sample to the MeOH-HCl mixture and mix
3. Heat at 60 °C for 10-15 minutes
4. Remove from heat and cool sufficiently to handle
5. Make basic with a saturated sodium bicarbonate solution (pH 8-9). Check pH with pH paper until desired pH is achieved.

6. Add approximately 0.5 mL of CH<sub>2</sub>Cl<sub>2</sub>, vortex, and let soak for one hour
7. Remove the CH<sub>2</sub>Cl<sub>2</sub> layer to a GCMS vial and fill with CH<sub>2</sub>Cl<sub>2</sub> to approximately 1 mL