

DRUG CHEMISTRY
DISCIPLINE
MANUAL

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1. Scope

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2. Normative References

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3. Terms and Definitions

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4. Management Requirements

4.1. Organization

1. Personnel Qualifications, Authorities and Responsibilities, and Duties

A. Chief Forensic Chemist

Qualifications:

The position requires the formal education equivalent of a bachelor's degree in chemistry; 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 Scientific Operations Director.

Authorities and Responsibilities:

The Chief Forensic Chemist is under administrative direction and is responsible for directing the activities of the Little Rock laboratory Drug Chemistry Section. 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 ASCLD/LAB International requirements by implementing lab wide policies and overseeing the section's quality assurance program.

Duties:

- i. Supervises a medium-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.
- ii. 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.
- iii. 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.
- iv. 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.
 - v. 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 methods.
 - vi. Compiles and interprets data obtained from analytical instruments, reviews and approves scientific forensic reports of section chemists, and writes conclusive scientific forensic reports.
 - vii. Provides classroom instruction for law enforcement officers at seminars and courses statewide on drug identification, collection of evidence, and clandestine drug laboratory investigations.
 - viii. 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.
 - ix. Performs related responsibilities as required or assigned.

B. Forensic Chemist Supervisor

Qualifications:

The position requires the formal education equivalent of a bachelor's degree in chemistry; 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 Scientific Operations Director.

Authorities and Responsibilities:

The Forensic Chemist Supervisor is under administrative direction and is responsible for directing the activities of the Hope Regional Laboratory. The Forensic Chemist Supervisor has the overall responsibility for the technical operations and the provision of the resources needed to ensure the quality of the laboratory operations. The Forensic Chemist Supervisor will have the appropriate technical training and technical experience in the drug section. The Forensic Chemist Supervisor will have regular contact with crime laboratory staff, frequent contact with law enforcement agencies and judicial officials, and limited contact with the public. The Forensic Chemist Supervisor ensures

compliance with ASCLD/LAB International requirements by implementing lab wide policies and overseeing the laboratory's quality assurance program.

Duties:

- i. Manages the work and personnel of the Hope Regional Laboratory by ensuring that work is completed timely and accurately, personnel are trained and developed, employees are informed and productive, needs of customers and employees are met, supplies are adequate, equipment is operational.
- ii. Coordinates with the Scientific Operations Director, Purchasing, Quality Assurance and Health & Safety Managers to ensure that the laboratory adheres to all prescribed quality assurance, safety, and security standards as well as technical protocols and agency policy.
- iii. Supervises a small 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.
- iv. Assists with developing laboratory policies and procedures, develops short and long-range operational plans for the laboratory, monitors operational activities by conducting staff meetings to disseminate information and reviewing and approving reports and compiles and submits statistical reports.
- v. 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.
- vi. 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.
- vii. 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 methods.
- viii. Compiles and interprets data obtained from analytical instruments, reviews and approves scientific forensic reports of section chemists, and writes conclusive scientific forensic reports.
- ix. Provides classroom instruction for law enforcement officers at seminars and courses statewide on drug identification, collection of evidence, and clandestine drug laboratory investigations.

- x. 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.
- xi. Serves as the site Health & Safety Manager, and Quality Assurance Manager.
- xii. Performs related responsibilities as required or assigned.

C. Forensic Chemist (Drug Chemist and Illicit Lab 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 12 week 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). This position is governed by state and federal laws and agency policy.

Authorities and Responsibilities:

- i. Process evidence suspected of containing controlled substance(s) submitted to the ASCL by law enforcement agencies.
- ii. Present expert forensic testimony in court on chemical analytical methodology used to analyze evidence and obtain results.
- iii. Participate in pretrial conferences and provide consultation to law enforcement and judicial officials on evidence collection, preservation methods and analysis results. Instruct law enforcement officials on proper methods of confiscating, preserving, and disposing of toxic chemicals, waste and equipment found in clandestine drug laboratories.
- iv. Verify the correct operation of scientific instruments and perform routine maintenance as needed. Prepare and verify standards and reagents according to established guidelines.
- v. Review current scientific literature. Study and validate new forensic analytical procedures and modify new and/or old procedures as necessary.
- vi. Attend and participate in professional meetings and seminars to keep abreast of new technologies and methods in chemistry.
- vii. Assist with training new laboratory staff in performing standardized laboratory test.
- viii. Perform related responsibilities as required or assigned.

The following are additional authorities and responsibilities outside of the basic authorities and responsibilities:

D. Quality Manager – Forensic Chemistry Section

- i. Ensures that instrument, balance, chemical/reagent and standard logs are recorded appropriately; prepares and records proficiency tests; maintains drug standard inventory.
- ii. Helps maintain and update the section's manuals and documents.
- iii. Monitors the section's practices for compliance with the section's SOP.
- iv. Ensures the validation of new technical procedures.
- v. Works with lab wide Quality Manager to seek ways to improve the quality system.

E. Health and Safety Officer – Forensic Chemistry Section

- i. Conducts monthly safety inspections and ensuring that proper practices and procedures are being followed in the section.
- ii. Maintains records of any safety incidents within the section.
- iii. Maintains a current copy of the section's MSDSs.
- iv. Works with the lab wide Health and Safety Manager to seek ways to improve the safety program.

F. Training Officer- Forensic Chemistry Section

- i. Makes sure reading material for new hires is relevant and up to date.
- ii. Helps maintain and update the training manual and forms.
- iii. Devises a training schedule for new hires.
- iv. Generates ideas for continued training for the section.
- v. Makes competency samples for new hires.
- vi. Works with Chief Forensic Chemist to seek ways to improve the training program.

G. Illicit Lab Chemist

- i. 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.
- ii. On call to aid law enforcement in safely dismantling illicit laboratories and collecting representative samples.

2. Deputies

A. Little Rock Laboratory

In the absence of the Chief Forensic Chemist, the analyst appointed by the Chief Forensic Chemist, or the analyst present with the highest seniority will serve as a deputy for key management personnel. All affected personnel will be notified.

B. Hope Regional Laboratory

In the absence of the Forensic Chemist Supervisor, the analyst present with the highest seniority will serve as deputy for Drug Chemistry and the technician present with the highest seniority will serve as deputy for Evidence Receiving. All affected personnel will be notified.

3. Communication

All employees will be notified of their responsibilities and expectations concerning the objective of the ASCL quality system and will be provided feedback on actual job performance through annual performance evaluations.

Supervisors will have routine meetings with their employees to convey information.

4. Organization and Management Structure

See organizational charts for Drug Chemistry at the Little Rock and Hope Regional Laboratories on the following pages. Each subordinate shall be accountable to only one immediate supervisor for each category of testing.

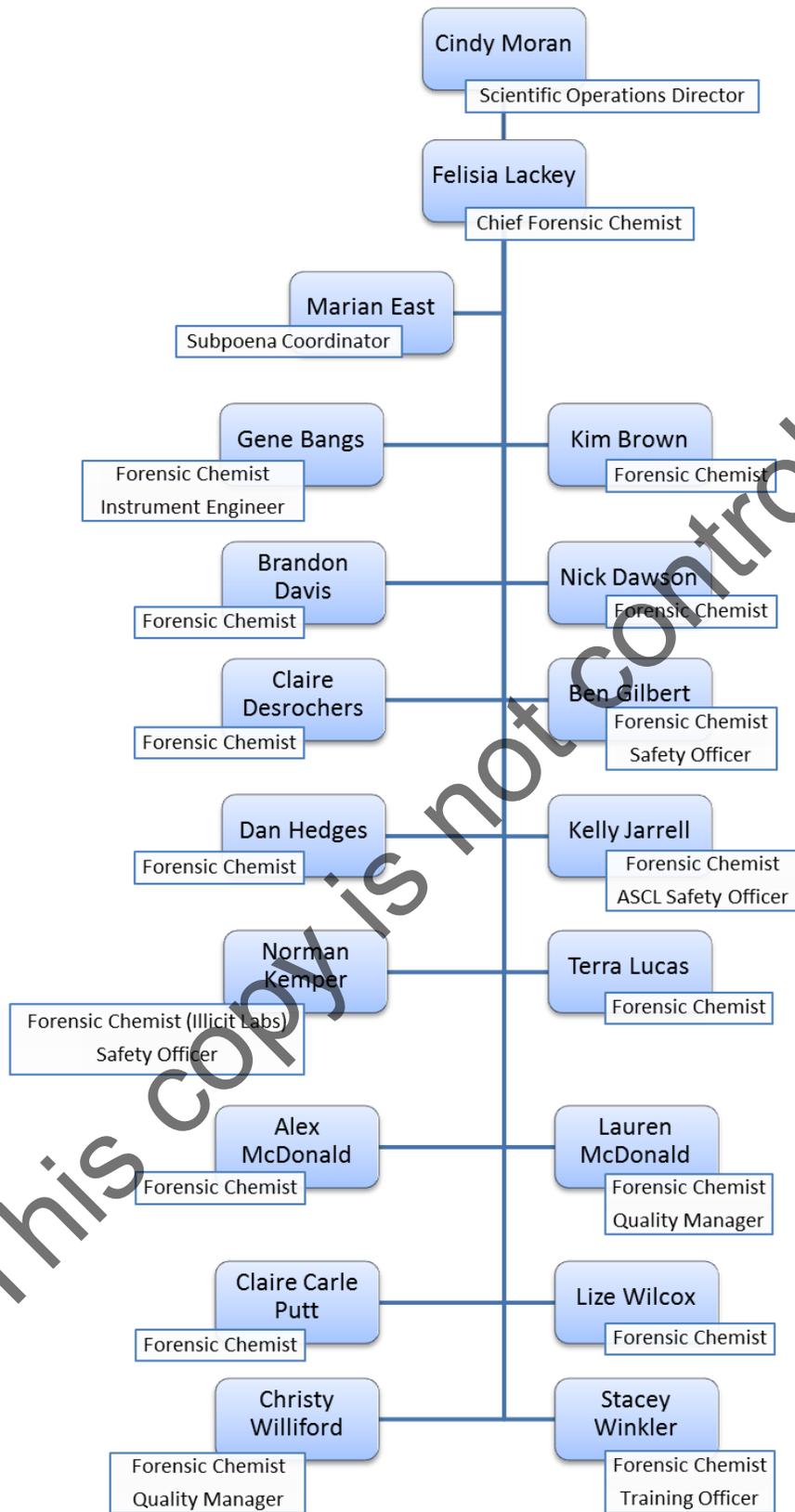
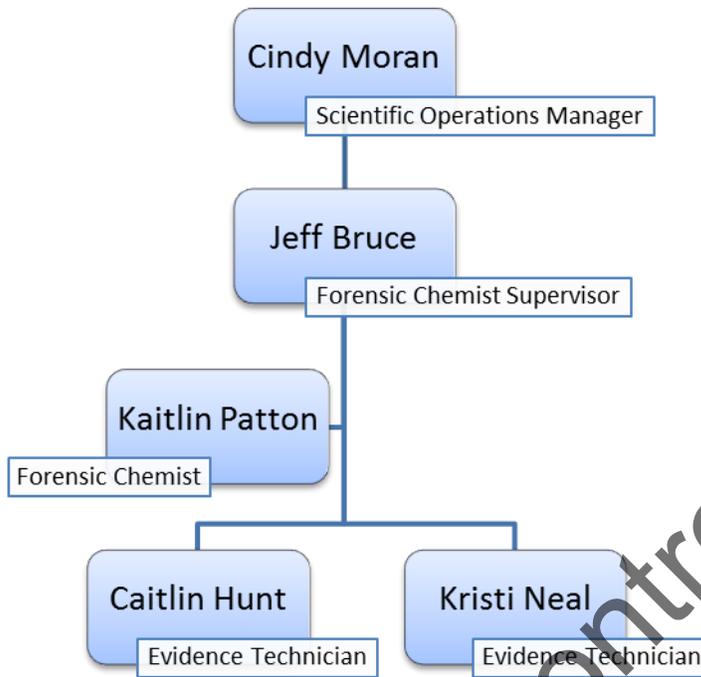


Figure 1 - Little Rock Laboratory Drug Chemistry



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Figure 2 - Hope Regional Laboratory Drug Chemistry

4.2. Management System

1. Mission

The mission of the Drug Chemistry discipline is to provide the highest quality scientific services and resources to the criminal justice community and others as authorized by law, through utilization of various scientific methodologies and instrumentation to analyze evidence¹ to identify controlled substances² and to provide information that may be pertinent to the investigation, charging and prosecution of individuals suspected of violating laws governing the possession, distribution or manufacture of controlled substances.

2. Documentation

This manual supports this mission by outlining the management, technical and quality assurance systems for the Drug Chemistry discipline. The manual governs operations in two distinct operational units (Little Rock Forensic Chemistry and Hope Forensic Chemistry), at two separate laboratory locations (Little Rock and Hope), and any sampling and analysis activities that may occur off-site. The supervisors for the two operational units jointly prepare, approve, and review this manual. However, chemists are only accountable to the supervisor who has responsibility for the work product of the operational unit in which they are performing work.

This manual should be considered a living document. All staff governed by these requirements should constantly review them. Policies and procedures may become obsolete or outdated and may require revision, replacement, or elimination. This manual will be updated as changes or additions occur.

The discipline mission is also supported by a *Drug Chemistry Training Manual* (DRG-DOC-02³) and a variety of forms. Official, controlled versions of these documents are available in Qualtrax.

¹ Evidence is defined as items submitted by law enforcement agencies to serve the purpose stated above.

² Controlled substance refers to drugs and/or chemicals placed on the Controlled Substance Act under federal law and Arkansas Code, Title 5, Chapter 64, Controlled Substances.

³ Version 02A contains readings from materials specific to the LR lab and version 02B contains those specific to Hope.

3. Deviations

Unforeseen circumstances may arise which require immediate deviations from the policies and procedures of this manual. In such situations, the request for exceptions to policy will be submitted in writing to the appropriate supervisor, or designee. The request must include an adequate description of the circumstances requiring the action, a statement of the proposed alternative policy and procedure, and the intended duration of the exception. The supervisor will maintain documentation of the approved policy exception.

4. Responsibilities

All Forensic Chemists are responsible for reviewing, knowing, understanding, and following these policies and procedures, to ensure quality, integrity, and accuracy in the examination and analysis of evidence.

5. Quality Assurance System

Major objectives of the quality assurance system are:

- Monitor, on a routine basis, the examinations, and analyses of chemists by means of quality control standards and proficiency tests.
- Verify that all discipline protocols and procedures are within established performance criteria, that the quality and validity of the analytical data are maintained and that the raw data gathered provides a sound foundation for reliable conclusions.
- Ensure that problems are noted and that corrective action is taken and documented.

4.3. Document Control

This manual, the training manual, and quality assurance forms for the Drug Chemistry discipline are controlled to ensure that they are adequate, approved for use, and that only the current versions of the document are in use. This procedure provides instructions concerning the creation, revision and distribution of these controlled documents. The Quality Assurance Manager will maintain the official controlled documents and archived versions of all controlled documents in QUALTRAX.

1. Preparation

Internally generated documents should be prepared by personnel with adequate expertise in the subject. The detail of the document should be commensurate with the complexity of the activity and the background of the intended user of the document. The document must include enough detail and specificity to ensure that the activity conforms to quality specifications or expectations. The preparer of the document is responsible for:

- A. Ensuring that the documents are scientifically suitable for issue.
- B. Ensuring that the documents contain the required quality assurance elements (i.e., QC, measurement of uncertainty, traceability).
- C. Preparing the document in the proper format.
- D. Addressing or resolving corrections from reviewing authorities.

2. Review and Approval

Each new or revised internally generated controlled document is required to be reviewed and approved.

- A. Drug Chemistry Manual - reviewed and approved by Chief Forensic Chemist, Forensic Chemist Supervisor, lab-wide Quality Manager, Scientific Operations Director, and Executive Director.
- B. Training Manual – reviewed and approved by Chief Forensic Chemist, Forensic Chemist Supervisor, and lab-wide Quality Manager.
- C. Quality System Forms - reviewed and approved by Chief Forensic Chemist, Forensic Chemist Supervisor, and lab-wide Quality Manager.

3. Revisions

While the authorities listed above are responsible for performing an annual review of the Drug Chemistry Manual and Training Manual and making any corrections or additions as necessary, revisions to controlled documents may occur at any time necessary. All revisions will be carried out according to the lab wide manual requirements (see ASCL-DOC-01 4.3.2.2 b).

4. Archiving

The official archive of retired controlled documents is maintained in Qualtrax. All other copies preserved by the discipline, whether print or electronic, is for knowledge preservation only and have no official standing.

5. User Responsibilities

New employee training will include how to access the official controlled documents relevant to their job function, and the hierarchy and inter-relationship between those documents. It is the employee's responsibility to verify that they are using the current revision of any document. Individuals may print hard copies of internal documents for personal use, but these will be unofficial copies. When revisions to a controlled document are made all affected personnel will be notified.

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4.4. Review of Requests, Tenders, and Contracts⁴

1. General

The customer should be contacted with any questions related to the agency's request. Case-related discussions with the customer, evidence discrepancies, and contract amendments will be documented (*Agency Contact Form - ASCL-FORM-06*, e-mail, or equivalent) and placed in the permanent case record.

Before analysis begins, a review is conducted by the appropriate supervisor or analyst to determine if there is anything more specific about the request and to determine if the laboratory has the capability and resources to perform the services requested (i.e. adequate standards, controls and approved test methods).

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

Once the submission sheet is accepted by the laboratory, any request for service submitted by a customer agency, either verbal or in writing, serves as a contract for service employing testing methods as described in section 5 of this Manual.

2. Review

During evidence processing, chemists will make sure that the evidence submitted matches the items listed on the contract. If the evidence matches the contract, the case is analyzed. If the evidence does not match the contract, the analyst shall notify the investigating officer, submitting officer, or property officer of the submitting agency. Contact with the agency, and a list of any discrepancies discovered by the analyst between actual and listed evidence will be documented. All attempts will be made to contact the officer to resolve the matter.

The Medical Examiner Section is considered an internal customer of the Little Rock Drug Section. The Evidence Report generated through JusticeTrax LIMS-plus will serve as the submission sheet for all evidence submitted by the Medical Examiner's office. The same process described above will be utilized to review requests from the Medical Examiner.

⁴ For definitions of Request, Tender and Contract, see section 3 of the ASCL Quality Manual (ASCL-DOC-01).

3. Deviations

When the customer agrees to the contract, the customer agrees that the ASCL may make deviations as deemed necessary. However, the customer will be notified (e.g. iResults, phone call email, etc.) if a request is cancelled, resulting in no analysis being performed.

4. Amendments

If a contract must be amended after work has begun, contact with the agency, and a list of amendments will be documented. All affected personnel shall be notified.

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4.5. Subcontracting of Tests and Calibrations

There are no additions to the lab wide quality manual.

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4.6. Purchasing Services and Supplies

If a material or service must meet certain specifications in order to properly function in testing, these items and the required specifications (i.e. manufacturer, type, grade or other technical data relevant to the supply or service) will be communicated to the Procurement Section through an External Supply 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 Drug Chemistry Discipline, the critical consumables are:

- Certified standards/reference materials
- PFTBA (perfluorotributylamine) GC-MS tuning compound

In the Drug Chemistry Discipline, the critical supplies are:

- Certified reference weight (for balance adjustment)
- Polystyrene reference materials (various forms) for FTIR performance verifications
- SUS reference material for XRF performance checks

4.7. Service to the Customer

There are no additions to the lab wide quality manual.

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4.8. Complaints

There are no additions to the lab wide quality manual.

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4.9. Control of Non-conforming Testing

There are no additions to the lab wide quality manual.

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4.10. Improvement

The Drug Chemistry Discipline strives to continually improve the effectiveness of its management system. To this end, the following activities are planned:

- An annual review of the management system
- Annual internal or external assessments
- A consideration of employee suggestions
- Evaluation of our work product through full technical and administrative review of all case files
- Evaluation of any received customer survey comments

Any employee may suggest changes to this manual by presenting the suggestion for evaluation, preferably in writing, to any of the discipline supervisors.

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4.11. Corrective Action

There are no additions to the lab wide quality manual.

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4.12. Preventive Action

There are no additions to the lab wide quality manual.

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4.13. Control of Records

1. General

A. Storage and Retention

Case files will be retained by the Arkansas State Crime Laboratory in either physical or electronic form. The Arkansas State Crime Laboratory is currently using the JusticeTrax LIMS-plus software program. All case documentation will be stored electronically. Once reviewed, this electronic version is the official case record.

Historical non-electronic case files for the discipline are stored in limited access areas to keep them secure and confidential.

Quality records, such as reagent and chemical logs and training records, will be readily accessible to employees whether in physical or electronic form.

B. Confidentiality of Records

See ASCL Quality Manual 4.13.1.3.

2. Technical Records

A. Retention

Case records are stored indefinitely. Quality records (e.g., logbooks) are stored for at least one full ASCLD-LAB-International accreditation cycle (i.e., four years). The following items are retained for at least fifteen years:

- Proficiency test records
- Corrective action documentation
- Assessment records
- Training records
- Continuing education documentation
- Court testimony monitoring records

B. Examination Records

Examination records are any records generated by the analyst/examiner for a case file (e.g. notes, worksheets, photographs, spectra, printouts, charts and other data). Examination records that are essential for the evaluation and interpretation of the data must be stored in the appropriate folder within the 'Request' folder in the JusticeTrax LIMS-plus case file.

- i. Observations, data, and calculations will be recorded at the time they are made and shall be identifiable to the specific task.
- ii. Examination records will reflect the date(s) of examination consistent with the requirements below in section 5.4.2.
 - The date of case note creation (see 5.4.2.2.E.ii) is the date that testing started.
 - The 'Date of Report' displayed on the 'Report of Laboratory Analysis' (i.e. the date for the request for analysis is marked 'Draft Complete' in LIMS-plus) is the date that testing ended unless documentation in the notes indicates otherwise.
- iii. Corrections to examination records will be consistent with the requirements in section 5.4.2.
- iv. Amendments to examination records will be handled according to lab-wide policy.
- v. The use of abbreviations will be consistent with the requirements in section 5.4.2.

C. Administrative Records

All other records contained in the case file will be considered administrative records and will be stored in the 'Case Images' folder.

4.14. Internal Audits

There are no additions to the lab wide quality manual.

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4.15. Management Reviews

There are no additions to the lab wide quality manual.

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5. Technical Requirements

5.1. General

1. Reagents, Chemicals, and Controls

The following rules shall be followed for reagents, chemicals, and controls:

- The quality of all chemicals purchased for use in the Drug Chemistry Discipline 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.
- 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 and standards used.
- Each analyst must ensure that the controls, reagents, or chemicals used in their analysis are of satisfactory quality.
- 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.

2. Preparation, Verification, Documentation and Labeling

A. General

- i. Specific reagent/chemical formulations (which may be scaled up or down depending on need), verification procedures, and lot documentation are located in the *Reagent Preparation and Chemical Preparation Logbooks*.
- ii. Reagents that are not routinely prepared will be subjected to QC testing appropriate for the reagent and recorded in the *Reagent Preparation Logbook*. The methods of preparation and verification must be recorded in the *Reagent Preparation Logbook*; otherwise all requirements are the same as those for routine reagents. However, the preparation of non-routine reagents for one time use may be documented (formulation, lot numbers of materials used and if applicable reference material(s) and results of any QC testing) in laboratory case notes and any excess reagent discarded after use.

- iii. Glassware used in the preparation of reagents, standards and controls should be clean. Volumetric flasks and pipettes used in the preparation of standards for quantitative work will be ASTM Class A.
- iv. Water used for aqueous preparations should be distilled or deionized.
- v. 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 make sure any excess from the prior batch is discarded.

B. Chemicals/Reagents

i. Purchased Chemicals/Reagents

Verification: Routine chemicals (solvents, acids, bases, KBr etc.) and reagents will be purchased of appropriate quality and used as received.

Labeling: Original containers of chemicals/reagents will be labeled with the lot number, expiration date (if applicable), date and initials received, and date and initials opened. Secondary containers to which chemicals are transferred should be marked with the content's identity and lot number.

ii. Prepared Chemicals

Preparation: Formulations for preparing routinely used chemicals are located in the *Chemical Preparation Log*. 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 standards) are not normally subject to additional QC measures.

Labeling: Containers of prepared chemicals will be labeled with the chemical's identity, preparer's initials, date of preparation and the expiration date. TLC systems will only be labeled with the chemical's identity.

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

iii. Prepared Reagents

Preparation: Formulations for preparing routinely used reagents are located in the *Reagent Preparation Logbook*. The actual amount of reagent made, the lot numbers (if applicable) of all materials used and the preparer's initials will be recorded in *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.

Labeling: Reagent containers must be labeled with the reagent's identity, preparer's initials, the date of preparation, expiration date, and the verifier's initials.

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

C. Controls

When controls are deemed applicable for a particular testing technique, their use and documentation are specified as part of the technique's description in section 5.4.5.

5.2. Personnel

1. Training Program

Each Forensic Chemist, regardless of prior training or experience, must complete a training program prior to assuming casework responsibilities. Normally the training is completed over a twelve week period. For analysts with prior experience, this training may be truncated (length, content, or both) with the approval of the employee's supervisor and the Scientific Operations Director. However, competency testing is always required regardless of prior training and experience.

The training program is detailed in the *Drug Chemistry Training Manual* (DRG-DOC-02⁵). Among the contents of this training are:

- Health and safety requirements
- Laboratory policies and procedures
- Instrumentation theory and practice
- Evidence handling and sampling procedures
- Analytical techniques and instrumentation
- Ethics in Forensic Science
- Criminal/civil law procedures
- Moot court
- Quality system requirements
- Interpretation and reporting
- General knowledge of Forensic Science
- Competency testing

Records will be maintained which document what training has occurred, and the evaluation(s) of that training. A written test will occur to document the trainee's knowledge of the subject material.

2. Employee Development Program

There are no additions to the lab wide quality manual.

3. Job Descriptions

There are no additions to the lab wide quality manual.

4. Competence Documentation

There are no additions to the lab wide quality manual.

⁵ Version 02A contains readings from materials specific to the LR lab and version 02B contains those specific to Hope.

5. Technical Personnel Qualifications

A. Education

Forensic Chemists shall possess a baccalaureate, or an advanced degree, in a natural science or closely related field.

B. Competency Testing

For all analysts and technical support personnel that generate analytical results, a competency test shall include, at a minimum:

- Examination of sufficient unknown samples to cover the anticipated spectrum of assigned duties and evaluate the individual's ability to perform proper testing methods;
- A written report to demonstrate the individual's ability to properly convey results or conclusions and the significance of those results/conclusions.

The unknowns are intended to mimic typically encountered exhibits. They encompass a range of analyte classes. It is not necessary to include every test method in the competency test, but commonly-performed test methods will be represented.

6. Literature

The Drug Chemistry discipline 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.

5.3. Accommodation and Environmental Conditions

1. Testing Environment

If environmental conditions are such that the validity or reliability of analytical results could be jeopardized, testing will be stopped until those conditions can be remediated.

2. Security

A. Little Rock Laboratory

The Forensic Chemistry section is in close proximity with the Toxicology and Trace Evidence Sections. The Forensic Chemistry Section is designed so that there is effective separation between neighboring areas in which there are incompatible activities.

Forensic Chemists, Toxicologists and Physical Evidence Analysts have security fob access to the Toxicology and Forensic Chemistry sections at all times. The shared access is due to the physical layout of the laboratory. Access is restricted for other members of the laboratory except those authorized by the Executive Director.

Each Forensic Chemist has lockable areas to store their 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 in the office of the Chief Forensic Chemist. 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 standards will be conducted as needed.

B. Hope Regional Laboratory

Overall security for the laboratory is detailed in the *ASCL Quality Manual* (ASCL-DOC-01). The Forensic Chemists and Evidence Technicians employed at the ASCL - HRF have access to the laboratory analysis area at all times. The shared access is due to the small staff size and the recognition that an Evidence Technician may be required to respond to an emergency in the laboratory analysis area.

Each Forensic Chemist has lockable storage areas for evidence in their possession. The Forensic Chemist Supervisor also has access to these storage areas.

Reference materials (i.e. drug standards) are stored in a locked filing cabinet in the chemical storage room. Keys to the filing cabinet are assigned to Forensic Chemist Supervisor and Forensic Chemist. The Forensic Chemistry Chief may designate other personnel as needed.

3. Health and Safety Program

Documented in the *ASCL Health and Safety Manual* (ASCL-DOC-08).

This copy is not controlled.

5.4. Test Methods and Method Calibration

5.4.1. Validations

There are no additions to the lab wide quality manual.

This copy is not controlled.

5.4.2. Case Notes

Chemists will create a set of contemporaneous case notes for each case request they analyze. Notes may be handwritten or typed. This section outlines content that must be documented in these case notes. Deviations from these guidelines must have the approval of the supervisor or designee.

1. 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:\SOP clarifications\forensic chemistry abbreviation definition list. A forensic chemist can add to this list at any time by defining a new abbreviation on the unsecure Abbreviations To Add list that can be found on S:\Abbreviations To Add. A Forensic Chemistry supervisor 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.

2. Requirements for Notes and Observations

- A. Handwritten notes and observations must be in ink. However, pencil may be appropriate for diagrams or making tracings.
- B. Nothing in the handwritten notes will be obliterated, erased, or deleted. Any corrections made to handwritten notes will be made by an initialed, single strikeout (so that what is stricken can still be read). Correction fluid or correction tape may not be used.
- C. Both the chemist's and trainee's handwritten initials must be present on each page of the case notes in cases in which a trainee assisted the chemist. Electronic equivalent can be substituted for chemist's handwritten initials.
- D. Additional notations, including interlineations, made in case notes must be initialed by the person making the additions.
- E. Each page of the case notes must include...
 - i. The unique ASCL case number (YYYY-00000),

- ii. the date(s) the notes were taken on (Should the sampling of a case take longer than one day, it should be properly noted which day the sampling was resumed.),
 - iii. and page number.
- F. The total number of pages must be documented by either using the method of "page 1 of x" or by including a statement that the case notes consists of x number of pages.
- G. Every evidence item should have an adequate description explaining the appearance of the item and its packaging. The description should be detailed enough so that the chemist could identify the evidence based only on their notes.
- H. Most items which are tested are required to have a measurement of their amount taken before and after sampling. These initial and reserve measurements must be documented in the case notes (see section 5.4. - Measurements for specific procedures).
- I. If sampling⁶ takes place, a reference must be made to the sampling plan or sampling procedure that was used.
- J. During testing, the chemist must document in their case notes...
- i. the tests performed,
 - ii. the date on which the tests were performed unless supporting examination documentation is marked with the testing date,
 - iii. and the results of the tests.
- K. A description of any solvent extraction procedures used on an item must be documented.
- L. Any other notations required for an individual testing technique are described in the appropriate subsection of Section 5.4.
- M. After the case notes are completed and before the case request status is marked 'Draft Complete' in LIMS-plus, the case notes must be imaged into the electronic case file.

⁶ See section 5.7

3. Requirements for Supporting Examination Documentation

Images of all other examination records for a request 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 LIMS-plus.

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5.4.3. Testing Requirements

1. Qualitative Analysis

A. General Specificity for the Results of Common Testing Techniques

Category A	Category B	Category C
Infrared Spectrometry (IR)	Gas Chromatography (GC)	Color Tests
Gas Chromatography/Mass Spectrometry (GC/MS)	Thin-Layer Chromatography (TLC)	
Energy Dispersive X-Ray Fluorescence (EDXRF)	Pharmaceutical Identifiers	

B. Quality of Test Results

Criteria for considering a test to be positive or negative for a compound will be addressed in each test's technique description (see section 5.4.5. and subsections). Criteria for an 'Indicative'⁷ result are provided for some testing techniques.

The results of category 'C' tests are indicative of the presence or absence of various drug classes and/or organic functional groups. The particular color test(s), if any, that are used by the chemist are usually indicated by the type of sample. Category 'C' tests normally serve as another aid for chemists in their determination of which items should be selected for analysis and to help plan future testing of test samples.

C. Minimum Testing of Exhibits

i. Minimum Tests 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 testing⁸. One of these tests must be from category 'A'; the second test may be from category 'A' or 'B', but not category 'C'. If only two tests are performed on the item, being a combination of one category 'A' and one category 'B' test, GC/MS (with a broad oven temperature program) is the preferred category 'A' test.

⁷ Indicative results are generally observations or data not meeting all the criteria for a positive result but may provide the chemist with investigative information to direct further testing.

⁸ i.e. In order to report results other than "not tested," "element(s)" name, or "identified as drug" on the *Report of Laboratory Analysis*, the minimum testing requirements per exhibit listed in this section must be met.

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, etc.) when selecting which compound to identify.

- ii. Minimum Tests per Exhibit: Exceptions
 - a. Items that test positive for tetrahydrocannabinol (THC) or marihuana (see this section 1.D.ii.b,& c.) are normally excluded from further testing.⁹
 - b. Samples taken for elemental analysis by EDXRF may be excluded from further testing.
- iii. Minimum Tests per Exhibit: Additional Requirements
 - a. If a controlled or penalty compound is indicated or detected by the minimum testing of exhibits, proceed to this section 1.D.i, ii, & iii. to determine if the minimum requirements for identifying the compound have been met.
 - b. If no controlled or penalty compound is indicated or detected by the minimum testing of exhibits, the Forensic Chemist may discontinue testing. Proceed to this section 1.D.iv. to determine if the minimum requirements for identifying a non-controlled compound have been met.

D. Minimum Testing For Each Element or Compound Identified

i. Controlled or Penalty Compounds

For each controlled compound identified in an item, the analyst should have as a minimum, two positive tests for that compound. One of these tests must be from category 'A'. If only two positive tests are performed, the second test may be from category 'A' or 'B', but not category 'C'.

ii. Controlled or Penalty Compounds: Exceptions

⁹ TLC plates run for the analysis of items suspected of being THC residue or marihuana must have their baselines over-sprayed with acidified iodoplatinate after their development with Fast Blue BB. If the visualization of an item with acidified iodoplatinate indicates (by comparison to the standard usually on or near the baseline) that another controlled substance may be present, further testing (GC/MS analysis at a minimum) is required.

- a. Positive tetrahydrocannabinol (THC) identification requires a positive result for THC from Gas Chromatography/Mass Spectrometry (GC/MS) analysis and positive results from any one of the following tests:
- qualitative gas chromatography (positive retention time match for THC),
 - thin-layer chromatography (for THC),
 - modified Duquenois-Levine (for cannabis.)
- b. Positive marijuana identification requires a positive microscopic examination of the plant material for cystolithic hairs and positive results from any two of the following tests:
- thin-layer chromatography (for THC),
 - modified Duquenois-Levine (for cannabis),
 - qualitative gas chromatography (positive retention time match for THC),
 - Gas Chromatography/Mass Spectrometry (GC/MS) (for THC)
- iii. Controlled or Penalty Compounds: Additions
Some isomers may only be differentiated, or a compound's salt/base form determined, by infrared spectrometry. If differentiation between stereoisomers (e.g. pseudoephedrine and ephedrine) is desired¹⁰, or salt/base determination is necessary¹¹, infrared spectrometry testing must be conducted.
- iv. Elements & Non-controlled Compounds
- a. The analyst may reach a positive conclusion on the presence and identity of elements in an exhibit based on results from X-Ray Fluorescence testing.
- b. The analyst may reach a conclusion that lithium is indicated in an exhibit based on the following tests:
- A positive or indicative result for lithium hydroxide or lithium carbonate by IR testing,
 - results of a flame test consistent with lithium,

¹⁰ Results of the IR testing must be either positive or indicative for pseudoephedrine or ephedrine. This requirement is waived for positive identification of pseudoephedrine or ephedrine in tablets or capsules which have been pharmaceutically identified to contain pseudoephedrine or ephedrine. (i.e. it is permitted to report pseudoephedrine or ephedrine based on a positive pharmaceutical identifier combined with a positive result from mass spectrometry.)

¹¹ 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 spectrometry, so that if possible, the cocaine form may be determined for reporting.

- c. The analyst may reach a positive conclusion on the presence and identity of a non-controlled substance based on one positive category 'A' test.
- d. The analyst may reach a conclusion that ammonia is indicated in an exhibit based on the following tests:
 - An indicative result for ammonia by IR testing,
 - and results of a Nessler's color test consistent with ammonia.

2. Quantitative Analysis

The Little Rock and Hope laboratories have quantification procedures in place for amphetamine and methamphetamine. State of Arkansas penalties for controlled substance offenses, when based on the weight, are based on the entire weight of a substance regardless of drug purity. It is the Agency's policy to not provide quantitative testing as a normal part of controlled substance analysis.

Federal guidelines allow for sentencing based on whether the actual drug weight or mixture weight results in the higher penalty level for PCP, amphetamine, methamphetamine, and oxycodone. The laboratory will honor requests for quantification of amphetamine or methamphetamine, but only in cases being prosecuted federally.

3. Tampering Analysis

Samples in this category may require the analyst to rely on their training and experience as a chemist to develop a testing procedure. No procedure manual could encompass methods for every tampering case analyzed. Good scientific principles and a logical analysis scheme are applied to those evidence types.

5.4.4. Measurements

Most items which are tested are required to have a measurement of their amount taken before and after a portion of the item is removed for testing. These initial and reserve measurements (numerical portion, and units) should be neatly recorded in the case notes. The section will assume weights recorded in case notes are net weights unless designated otherwise. It is the responsibility of the chemist working the case to clearly label gross weights, calculated net weights, or counts by weight as such in their notes.

1. Weighing

A. Weighings to Perform

All items that will be tested¹⁵ will have their initial net weight measured before a sample is collected unless the item is a residue in the chemist's opinion. An item's net weight shall be acquired directly. Even if analytical testing will not be done, measure an initial combined net weight of all tablets and capsules if they have been pharmaceutically identified to contain a controlled substance or penalty drug¹⁶. If an item does not exceed the minimum weight specification for the balance being used¹⁷ the item will be weighed on a balance designed for a lower weight range. If the item does not exceed the minimum weight specification for the analytical balance, the measured weight will be documented in the notes and reported as 'less than 10mg' on the report, or some similar terminology. After a sample is collected, the item's reserve net weight will be measured¹⁸.

<i>Balance Type</i>	<i>MoU¹²</i>
Analytical	0.0010 g
Toploader	0.2 g
LR Bulk ¹³	2 g
Hope Bulk ¹⁴	0.01 kg
* for single weighing by balance type.	

Measurements will be performed using a verified balance (see section 5.5.2.) suitable for the item's size. The measurements will be made in grams or kilograms and should be recorded in the case notes exactly as displayed on the

¹² The MoU calculations and budget are located on the drug S:\SOP Clarifications\MoU documents.

¹³ Mettler TXS32000L

¹⁴ Ohaus ES50L

¹⁵ This requirement shall be waved for evidence involved in manufacturing charges. The chemist will use their training and experience to determine if a sample should be weighed.

¹⁶ Sealed containers (e.g. sealed blister packs) of penalty tablets or capsules are excluded from the weight measurement. The tablets or capsules will be counted only.

¹⁷ i.e. bulk 60 g, toploader – 5.0 g, analytical - 0.0100 g.

¹⁸ In situations where the sample mass is negligible compared to the mass of the item, the mass of sample taken may be satisfactorily substituted.

balance. The measurement of uncertainty (MoU) for a single weighing on each type of balance is given in Table 5.4.4.-1

When a chemist processes multi-item populations¹⁹ and does not test all the sub-items²⁰, several weighings are required. The gross weight of the entire population should be measured at the outset, initial and reserve weights will be measured for each sub-item tested, and the gross weight of all untested sub-items will be measured at the finish. It is the chemist's responsibility that these weights are clearly labeled and there is no ambiguity to the relationship between the measurement and the items measured.

B. Calculation(s)

i. Total Net Weight & MoU for Multiple Summed Items

For a single exhibit containing multiple items or consecutive multiple exhibits with identical test results and weighed on the same balance, the individual weights as recorded may be summed.

If multiple weighings are summed, the total uncertainty in the calculated amount depends on the number of measurements summed (n) and the uncertainty associated with each weighing operation (u). The total MoU for the summed weighings can be calculated using Equation 5.4.4.-1²¹.

EQUATION 5.4.4.-1 *Calculating MoU for Multiple Summed Items*

$$U = \sqrt{n \times u^2}$$

U = total uncertainty, n = number of items summed, u = uncertainty in a single weighing

ii. Calculated Net Weights & MoU

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 Equation 5.4.4.-2.

EQUATION 5.4.4.-2 *Calculated Net Weights*

$$M_{net} = M_{gross} \times \left[\frac{\sum_{n=1}^n m_n}{(M_{gross} - \mu_{gross})} \right]$$

M_{net} = net weight of the entire population (calculated), M_{gross} = gross weight of the entire population, n = number of sub-items tested, m = net weight of each tested sub-item, μ_{gross} = entire gross weight of untested sub-items.

The measurement of uncertainty for the

¹⁹ Other than tablets or capsules.

²⁰ Always true if the sampling plan is used.

²¹ Pre-calculated uncertainties for n values up to 50 can be found on the Summary sheet of the budget spreadsheet: S:\SOP clarifications\MOU documents\MOU Budget Form

calculated net weight is determined by using spreadsheet DRG-DOC-03.

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2. Counting

A. Requirements for Count Measurement

In addition to weighing, solid dosage forms (e.g. tablets, capsules, suspected LSD on blotter paper squares or sugar cubes) will be counted.

B. Calculation(s)

For exhibits containing more than 160 pharmaceutical tablets or capsules, the analyst may calculate the total number of tablets or capsules present in the exhibit using Equation 5.4.4.-3.

EQUATION 5.4.4.-3 Total Tablets/Capsules by Weight

$$n_{Calc} = \frac{m_{Total} * n_{Count}}{m_{Count}}$$

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.

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}) by...

$$n_{Calc} = (241.8136 * 160) / (10.2121) = 3788.66 \text{ tablets}$$

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

5.4.5. Testing Techniques

5.4.5.1. Introduction

This section describes the testing techniques commonly utilized for the analysis of evidence exhibits in the Drug Chemistry Discipline. Strategies for analyzing specific types of evidence exhibits for suspected drug(s) are addressed in the training manual.

The description of testing techniques presented in this section should not be considered to exclude the use of other verified or published techniques. Chemists are encouraged to consult their peers or scientific literature on an as needed basis.

The chemist must be able to make modifications in basic analytical techniques to accommodate changes in the type of evidence exhibits received and changes to the legal code. New techniques may be incorporated in this manual as they are developed and validated.

It is acceptable for laboratory procedures to specify where specific record components (e.g. spectra of standards or calibration documentation) are maintained without a reference to the location of these records in the case file.

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

5.4.5.2. Color Testing

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.

1. General Method

- 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 your observations in the case notes.

NOTE: Adding the appropriate reagent to the well plate depression before adding the sample can effectively act as a blank.

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.

A. Single Reagent Color Tests

- i. Marquis
- ii. Ferric Chloride
- iii. *p*-dimethylaminobenzaldehyde (PMBA)

B. Multiple Reagent Color Tests

- i. Sodium Nitroprusside
In step ❶ add Nitroprusside "A", add Nitroprusside "B" in step ❸.
- ii. Cobalt Thiocyanate/Stannous Chloride
In step ❶ add Cobalt Thiocyanate, add Stannous Chloride in step ❸.

C. Other Color Tests

- i. Nessler's — testing for $\text{NH}_3(\text{g})$:
 - a. Place sample in a well plate depression,
 - b. add base if necessary,

- c. apply Nessler's reagent to a microscopic slide and invert over the depression,
 - d. examine the reactants for any changes,
 - e. and record in the case notes.
- ii. Modified Duquenois-Levine
- This test is appropriate for testing vegetation or smoking device extracts for cannabinoids. Solvents normally used for the extraction include pet ether, hexanes, ligroin etc.
- a. Transfer a portion of the extract to a new labeled test tube. Heat the test tube to reduce the solvent volume if necessary.
 - b. Add approximately 1 mL of Duquenois-Levine reagent.
 - c. Add approximately 1 mL of concentrated hydrochloric acid (HCl).
 - d. Agitate the solution and observe any color change.
 - e. Add approximately 1 mL of methylene chloride to the solution and agitate.
 - f. Observe the color of the bottom layer and record in the case notes.
- iii. Flame Test
- When a metal salt is introduced into the flame of a Bunsen burner, the metallic ion produces characteristic color in the flame.
- a. In a safe area, ignite a Bunsen burner.
 - b. Obtain a piece of nichrome wire with a loop in one end.
 - c. To clean the wire first dip the wire loop into dilute (~0.1M) hydrochloric acid and then into deionized water.
 - d. Heat the wire loop in the Bunsen burner flame until the wire begins to glow. Repeat steps c & d until no color is observed in the flame.
 - e. Dip the looped end of the wire into a sample²². 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.

²² 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.

5.4.5.3. Thin Layer Chromatography (TLC)

Scope: 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.

1. Solvent System Selection & Preparation

A wide variety of solvent systems are described in TLC literature. TABLE 5.4.5.3.-1 lists the most common solvent systems used, however the use of any published TLC solvent system is acceptable.

System	Makeup	Useful For...
Davidow	Davidow solution ¹ : 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 ² : Diethyl Ether (80:20)	Cannabinoids
Steroids	Methylene Chloride ³ : Ethyl Acetate (80:20) OR Methylene Chloride ³ : Methanol (90:10)	Steroids

¹ Ethyl Acetate: Methanol (85:10). ² Petroleum Ether or Ligroin may be substituted. ³ 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.

2. Technique

A. Sample Preparation

Solid samples are dissolved in an appropriate solvent. Liquid samples may be used as is or diluted in an appropriate solvent. Some samples may require an extraction procedure to remove interfering compounds.

B. Sample Application

- i. A line is drawn with a pencil parallel to, and ~2 cm from, the bottom of the TLC plate.
- ii. The samples are spotted on this line, called the origin, starting ~2 cm from the side of the plate and ~1 cm from each other.
- iii. The sample and standard spots are labeled uniquely. The chemist must be able to correlate the sample spot with the case and exhibit number.
- iv. The sample(s) and standard(s), in as small a volume of solvent as possible, are applied with a capillary tube.

- v. The sample solutions may be applied in aliquots, and dried before the plate is run.

C. Running the Plates

- i. 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.
- ii. Normally the plate is allowed to develop through a distance of 10-15 cm.
- iii. When the development period is complete, the plate is removed from the tank and allowed to dry before visualization.

D. Visualization Techniques

As most organic compounds are colorless, they must be made visible so that their relative retention values 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 TABLE 5.4.5.3.-2 below.

Visualization Technique	Useful For...	Comments
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 dependant.)
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
Ethanol: H ₂ SO ₄ (4:1)	Steroids	Heat plate after spraying

3. Precautions & Possible Sources of Error

A. Sample Preparation

- i. Poor choice of solvent resulting in low solubility of solute(s) of interest.
- ii. Sample solution is too dilute.
- iii. Samples containing interfering compounds may require an extraction or other clean-up procedure to remove the interference.

B. Sample Application

- i. Spot should be no more than ~4 mm in diameter or resolution will be lost.
- ii. The plate surface must not be cut or gouged by the applicator.
- iii. 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.

C. Running the Plates

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

D. Visualization

- i. 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.
- ii. Compounds may be present on the plate in small concentrations or ionic forms that may not be visible.

4. Data Interpretation

A. General

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

If a sample contains compounds with similar relative retention values 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.

B. Positive Results

A compound in a sample matches the relative retention value and visualization reaction(s) of a standard on the same plate.

C. Negative Results

No spots that match a standard are visible in the sample lane.

5. Notations Specific to This Test

A. General

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

- i. Type of solvent system used,
- ii. visualization technique(s) employed (e.g. UV, Ninhydrin, etc.),
- iii. the standard designation of any reference standard used to yield a positive result.
- iv. the date of the testing.

B. Positive Results

Any compound(s) meeting the criteria for positive results (as defined in 5.4.5.3. Part 4B) will be entered into the case notes by chemical name or an appropriate abbreviation.

C. Negative Results

Samples meeting the criteria for negative results (as defined in 5.4.5.3. Part 4C) will be entered into the case notes by either the designation "Negative" or "No spots."

5.4.5.4. Fourier Transform Infrared Spectroscopy (FTIR)

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.

1. Calibration & Maintenance

Refer to section 5.5.3.

2. Instrument Operation Parameters

Number of Scans	8
Resolution	4.000 cm^{-1}
Sample Gain	Auto
Scanning Range	4000-400 cm^{-1}
* These should be considered starting point values only and may be adjusted by the chemist depending on the type of information needed.	

3. Technique(s)

A. Transmission Experiments

i. Common Sample Preparation Techniques

a. Solid Phase Technique

An amount of sample is typically mixed with finely ground KBr using a mortar and pestle (~1:100 ratio of sample to KBr by weight).

A sample card (a paper/cardboard card with a hole in the center) is placed on a metal die, and the sample is placed in the sample card hole. The other metal die is placed on top of the sample card and placed in a hydraulic press. Approximately 15,000 psi is applied. The metal dies are removed from the press and the sample card is removed.

b. Vapor Phase Technique

A blank spectrum should 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).

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 cell is placed in the holder inside the sample chamber.

c. Liquid Phase Technique

There are two basic techniques that may be used to prepare the liquid sample to be scanned.

- Using salt plates: A blank spectrum should be acquired by placing two clean salt plates in the sample chamber before each liquid phase IR sample to ensure that the plates are clean (the salt plates can be cleaned by wiping the salt plates with a clean wiping paper). A small drop of the liquid sample is applied to a salt plate. A second salt plate is carefully placed on top of the first plate so that the sample evenly spreads out over the two plates.
- A sample card is placed on a metal die and KBr powder is placed in the sample card hole. The other metal die is placed on top of the sample card and placed in a hydraulic press. Approximately 15,000 psi is applied. A few drops of the liquid sample are placed on the KBr window.

ii. Running the Sample(s)

The prepared sample (i.e. sample card, vapor phase cell, salt plates) is placed in the holder inside the sample chamber.

The spectrum is acquired.

B. Reflectance (ATR) Experiments

i. Common Sample Preparation Techniques

Normally no sample preparation is needed to acquire infrared spectra of samples in Attenuated Total Reflectance (ATR) experiments.

ii. Running the Sample(s)

a. A blank spectrum should be acquired before any sample.

b. Solid Samples

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

c. Vapor Samples

N/A in ATR.

d. 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.

4. Precautions & Possible Sources of Error

A. Instrumental

- i. Verify that all necessary calibration checks have been done and that the instrument has passed each one.
- ii. Poor bench alignment which is characterized by:
 - a. For a background spectrum, the %T at 4000 cm^{-1} approaches zero,
 - b. and/or after a sample spectrum has been baseline corrected, the baseline still “rolls” (i.e. the sample peaks appear on top of a decaying sinusoidal wave.)

B. Sample Preparation

- i. Sample(s) are prepared in too dilute a form (IDEAL: The strongest peak will have an absorbance of at least 0.6.)
- ii. Sample(s) are prepared in too concentrated a form (IDEAL: The strongest peak will have an absorbance of no more than 1.2.)
- iii. To avoid damaging the hydraulic press, no more than ~20,000 psi should be applied.
- iv. Aqueous samples should not be used with salt plates because the plates could be damaged.
- v. Samples containing interfering compounds may require an extraction or other clean-up procedure to remove the interference.

C. Running the Sample(s)

- i. Unusual matches suggested by the software matching algorithm: Check which search libraries are selected.
- ii. The spectrum contains incompletely subtracted background peaks (e.g. H_2O absorptions at 3800 and 1600 cm^{-1} , and CO_2 absorptions at 2350 and 668 cm^{-1}): Collect a new background and re-run sample.

5. Data Interpretation

A. General

Identification of an unknown sample is based on comparing the sample's infrared spectrum with reference spectra. 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.

B. Positive Results

An acceptable blank will not be a positive or indicative match for a controlled substance or a common cutting agent.

The sample's infrared spectrum visually matches that of the reference standard spectrum. All peaks present in the reference standard spectrum are also present in the sample's spectrum and there are no peaks in the sample's spectrum which are not present in the reference standard spectrum (except for peaks incompletely removed by background subtraction or small peaks that can be explained by a change in resolution from standard spectrum to sample spectrum.)

C. Indicative Results

An infrared spectrum may be indicative of one or more compounds in the following instances:

- i. Any manipulated sample spectrum obtained by subtraction of one or more reference standard spectra from the original sample spectrum.
- ii. The reference spectrum best available match for the sample spectrum is of a mixture.
- iii. All peaks present in the reference standard spectrum are also present in the sample's spectrum and there are peaks in the sample's spectrum which are not present in the reference standard spectrum (except for peaks incompletely removed by background subtraction.)

D. Negative Results

The sample doesn't visually match any available reference standard spectrum.

6. Notations Specific to This Test

A. 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.

i. Case Notes

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

- a. Type of sample preparation and,
- b. acquisition technique employed (e.g. s, l, g),

ii. Instrumental 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 LIMS-plus. The ASCL case number, exhibit number, and date must be visible on the image.

B. Positive Results

Any compound(s) meeting the criteria for positive results, as defined in section 5.4.5.4. part 5.B., will be entered into the case notes by chemical name or an appropriate abbreviation.

C. Indicative Results

Any compound(s) meeting the criteria for indicative results, as defined in section 5.4.5.4. part 5.C., will 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.

D. Negative Results

Samples meeting the criteria for negative results, as defined in section section 5.4.5.4. part 5.D., 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) should be enclosed in brackets.)

5.4.5.5. Gas Chromatography (GC)

Scope: The methods in this document describe various techniques used to prepare samples and obtain gas chromatography spectra for both qualitative and quantitative sampling (methamphetamine and amphetamine only), precautions and possible sources of error, precision requirements, data interpretation, and notations specific to this test.

1. Qualitative Analysis

A. Calibration

Refer to section 5.5.4.

B. 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 (i.e. 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 (i.e. split, splitless, etc.), 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.

C. Technique

i. Sample Preparation

The sample(s) and any necessary standard(s) should be prepared at approximately the same concentration(s). The sample and standard should not be acidic or basic or contain any solid material. Blanks should be prepared with the same solvent(s) used to prepare the sample and standard respectively.

ii. Running the Sample(s)

Note: Two options follow for running samples. The options are only intended to reflect the relationship between samples and the standard(s) that they are being compared to. When multiple samples are run, the Chemstation sequence may actually be a mixture of the two options.

Option 1: If the chemist has n samples for qualitative testing with a variety of analytes of interest, blanks, standards and samples should be run by repeating the pattern $bsBS$ in the following sequence:

Option 1: $b_1s_1B_1S_1\dots b_ns_nB_nS_n$
where,
 b = a solvent blank preceding a sample,
 s = a sample,
 B = a solvent blank preceding a standard,
 S = a standard.

All members of the set $b_ns_nB_nS_n$ must be run under identical chromatographic conditions.

Option 2: If the chemist has n samples for qualitative testing all with the same analyte of interest, blanks, standards and samples should be run by the following pattern:

Option 2: $B_1S_1b_1S_1\dots b_ns_nB_2S_2$
where,
 b = a solvent blank preceding a sample,
 s = a sample,
 B = a solvent blank preceding a standard,
 S = a standard.

All members of the sequence must be run under identical chromatographic conditions.

D. Precautions & Possible Sources of Error

- i. The sample and standard solutions should not be acidic or basic or contain any solid material.
- ii. Samples and standards do not have approximately the same concentration.

E. Data Interpretation

- i. General

Option 1: The data generated by the sequence $b_ns_nB_nS_n$ will be considered a set and evaluated on the basis of the set's members only (i.e. the retention time (t_R) of s_1 will only be compared with the t_R of S_1 and never S_2, S_3 , etc.)

Option 2: All samples will be compared to both the standards which bracket the sequence.

The chromatograms of the blanks b_n and B_n must not contain any peaks (signal to noise ≥ 3) at the analyte(s) retention time(s). The analyte peak of the chromatograms for s_n and S_n must have a signal-to-noise ratio ≥ 10 .

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 standard, within the tolerances listed in TABLE 5.4.5.5.-1.

Retention Time	Tolerance
≤ 3 minutes	$\pm 2\%$ relative
> 3 minutes	$\pm 1\%$ relative

The relative retention time is calculated according to EQUATION 5.4.5.5.-1.

ii. Positive Results

Option 1: The calculated relative retention time of the sample s_n versus S_n is \leq the acceptable tolerance listed in TABLE 5.4.5.5.-1.

Option 2: The calculated relative retention time of the sample s_n versus both S_1 and S_2 is \leq the acceptable tolerance listed in TABLE 5.4.5.5.-1.

iii. Indicative Results

N/A

iv. Negative Results

The calculated relative retention time of the sample s_n versus S_n is greater than the acceptable tolerance listed in TABLE 5.4.5.5.-1 and/or the analyte peak of the chromatograms for s_n and S_n has a signal-to-noise ratio ≤ 10 .

F. Notations Specific to This Test

i. 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 relative retention time calculation(s), and reference standard designation(s) will be shown on the printouts and/or the case notes.

a. Case Notes

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

- Type of sample preparation, and
- the results of the relative retention time calculation.

b. Instrumental Printouts

Images of chromatograms (blanks, standards & 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 LIMS-plus. The ASCL case number and the exhibit number 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.

All chromatograms (blanks, standards, and samples) must be stored electronically in the case file. The ASCL case number, exhibit number, and date must be visible on the image (except standard blanks and standards). For runs utilizing a nonstandard method, the instrumental parameters (method) must also be stored electronically in the case file (Only one copy per method per case file is necessary. It must be treated as examination records).

ii. Positive Results

Positive test results should be recorded in the case notes in a manner similar to "Positive (+) retention time (t_R) match for *compound*."

iii. Indicative Results

N/A

iv. Negative Results

Negative test results should be recorded in the case notes in a manner similar to "Negative (-) retention time (t_R) match for *compound*" or "Negative (-), No peaks."

2. Quantitative Analysis: Amphetamine and Methamphetamine

A. Calibration

Refer to section 5.5.4.

B. Instrument Operation Parameters

Use TABLE 5.4.5.5.-2 to select the GC method associated with the compound that is being quantitated. The method will set the instrument parameters.

Analyte	GC Method(s) ¹ ♦
methamphetamine	meth_frnt, meth_back
amphetamine	amp_frnt, amp_back

¹ frnt and back refer respectively to the front and back GC injectors.

C. Technique

i. Sample Preparation

The quantitation procedure utilizes a solution of n-tridecane (i.e. C13) in methylene chloride as an internal standard (IS). Refer to the *Tridecane (C13) Internal Standard Solution* log sheet (DRG-FORM-28) for details on preparing this solution if an unexpired solution is not available. After obtaining the IS follow the steps listed in TABLE 5.4.5.5.-3 to correctly prepare the samples for quantitative analysis.

1	Weigh out a 20-100 mg portion ¹ of the sample ² to be quantitated and transfer to a screw-top vial.
2	Record the amount in the case notes.
3	Using a volumetric pipet and proper technique, transfer a 1 mL aliquot of the appropriate IS solution to the screw-top vial.
4	Add ~2 mL of strong base solution ³ .
5	Add ~5-12 mL of methylene chloride.
6	Cap the screw-top vial and vortex (~15 s) or shake (~1 min.)
7	Centrifuge the screw-top vial and/or dry the methylene chloride layer over Na ₂ SO ₄ .
8	Transfer a portion of the methylene chloride layer to an auto-sampler vial and cap.

¹ A larger portion (i.e. > 100 mg) may be necessary for extremely weak samples. ² The same procedure is used to prepare a check sample by substituting a known amount (10-100 mg) of a verified primary reference compound for sample in step **1** (see below, section 5.4.5.5. part 2.C.ii.). ³ Normally 10% KOH, NaOH, etc.

ii. Running the Sample(s)

Before running any samples, check the *GC Log Sheet* to determine if the calibration curve(s) that are to be used have been verified that day. If the calibration curve(s) that are to be used have not been verified, obtain and run an appropriate check sample. Evaluate the calibration curve by calculating the percent difference between the known amount of standard in the check sample versus that calculated from the calibration curve using EQUATION 5.4.5.5.-2.

$$\text{EQUATION 5.4.5.5.-2 Percent Difference}$$
$$\% = \frac{(\text{Amount}_{\text{weighed}} - \text{Amount}_{\text{calc}})}{\text{Amount}_{\text{weighed}}} * 100\%$$

If the percent difference is $\leq 5\%$, the calibration curve is verified for use and the chemist may run their sample(s). If the percent difference is $> 5\%$, the calibration curve is removed from service until error is resolved.

A solvent blank will be run before each sample using the same GC method for both.

Blank and sample aliquots are introduced into the GC by automated injection. A report showing the chromatogram and calculated compound amount is automatically generated by the ChemStation software at the conclusion of the run.

D. Precautions & Possible Sources of Error

- i. Improper pipetting technique.
- ii. Improper internal standard preparation.
- iii. Not enough base is added.
- iv. Compounds co-eluting with either the sample or internal standard
- v. No IS transferred to the screw-top vial.

E. Data Interpretation

i. General

The blanks must not contain any peaks (signal to noise ≥ 3) at the analyte(s) or internal standard retention time(s).

ii. Sample Precision Requirements

The calculated weight from the curve must be within the low point and high point on the curve.

The percent purity of each run will be calculated according to EQUATION 5.4.5.5-3 and truncated to three (3) significant figures.

EQUATION 5.4.5.5-3 *Percent Purity*

$$\% = \frac{\text{Amount}_{\text{calc}}}{\text{Amount}_{\text{weighed}}} * 100\%$$

The calculated compound amounts on the report are reported *as the hydrochloride salt*. It may also be necessary for the chemist to convert their answer to the base or a different salt form. See TABLE 5.4.5.5-4 for some common conversions.

- iii. Positive Results
N/A
- iv. Indicative Results
N/A
- v. Negative Results
N/A

Compound	Conversion	Factor
Amphetamine	Hydrochloride to Base	0.7876
	Sulfate to Base	0.7378
	Sulfate to Hydrochloride	0.9368
Methamphetamine	Hydrochloride to Base	0.8036

F. Notations Specific to This Test

i. General

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

The percent purity calculation(s) will be shown on the printouts or the case notes.

ii. Case Notes

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

- a. Type of sample preparation
- b. the sample amount(s) used
- c. the calculated percent purity
- d. standard designation of the internal standard

iii. Instrumental Printouts

Images of chromatograms (blanks, standards & 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 LIMS-plus. The ASCL case number, exhibit 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.

This copy is not controlled

5.4.5.6. Gas Chromatography/Mass Spectrometry (GCMS)

Scope: The methods in this document describe various techniques used to prepare samples and obtain gas chromatography/mass spectrometry spectra, precautions and possible sources of error, precision requirements, data interpretation, and notations specific to this test.

1. Calibration & Maintenance

Refer to section 5.5.5.

2. Instrument Operation Parameters

A. GC Parameters

Instrument operation parameters are only one factor in obtaining a 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 (i.e. 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 (i.e. split, splitless, etc.), 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.

B. MS Parameters

Qualitative data should always be collected in full scan mode with the high mass scanned exceeding the analyte's molecular weight by at least 10 amu. The MS must be re-tuned if the scan range is changed.

3. Technique(s)

A. Sample Preparation

Sample and Blank Preparation

The sample may be prepared by a solvent dilution or extraction and should not be acidic or basic or contain any solid material. The blank should be prepared with the same solvent used to prepare the sample.

B. Running the Sample(s)

A blank should be performed before each sample.

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.

4. Precautions & Possible Sources of Error

A. Instrumental

Instrument not calibrated.

B. Sample Preparation

The sample and standard should not be acidic or basic or contain any solid material.

Poor choice of solvent (low analyte solubility) or extraction scheme.

C. Running the Sample(s)

Co-eluting compounds.

Scan range may have to be adjusted.

5. Data Interpretation

A. General

An acceptable blank will not contain any peaks (signal to noise ≥ 3) whose mass spectrum is a positive or indicative match for a drug or common cutting agent.

Identification of unknown compound(s) in a sample is based on comparing the sample's mass spectrum with 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:

- i. A printout of the original full mass scan for the signal area.
- ii. A printout of the full mass scan for the subtraction area.
- iii. A printout of the subtraction results.

The electronic data files generated from each GC/MS run (samples and blanks) will be retained for a minimum of three (3) months post acquisition.

B. Positive Results

The mass spectrum of a peak in the sample's chromatogram visually matches that of the reference standard spectrum and all five (5) of the following criteria are met:

- i. The signal-to-noise ratio of the chromatographic peak is ≥ 10 .
- ii. The identified compound is not present in the preceding solvent blank.
- iii. If the reference spectrum shows a molecular-ion peak for the compound, the sample's mass spectrum must also contain the molecular ion peak.
- iv. All peaks present in the reference spectrum should be present in the sample's spectrum with the following exceptions:
 - a. Peaks in the reference spectrum that are below the scan limits set in the method parameters [NOTE: Scan limits for the method should be modified for samples suspected to contain compounds such as GBL or GHB, which have significant peaks outside the normal scan limits.]
 - b. Peaks in the reference spectrum that are higher in mass than the molecular-ion or the molecular-ion isotopic peaks (if applicable).
 - c. Low abundance ions (below 10% of the abundance of the base peak) may be absent unless the ion is also the molecular ion.
- v. There should not be any extra peaks in the sample's spectrum when compared to the reference spectrum with the following exceptions:
 - a. Low background peaks (below 10% of the abundance of the base peak) are ignored.
 - b. If the reference spectrum has a limited scan range, the sample spectrum should be compared to a different reference or a standard spectrum can be acquired for comparison.

C. Indicative Results

The mass spectrum of a peak in the sample's chromatogram is visually similar to that of the reference standard spectrum, but the sample's mass spectrum doesn't meet all of the criteria for a positive result.

D. Negative Results

The mass spectrum of a peak in the sample's chromatogram does not visually match any available reference standard spectra.

6. Notations Specific to This Test

A. General

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

i. Case Notes

The chemist will include the type of sample preparation in the case notes for each GC/MS test performed.

ii. Instrumental Printouts

Images of chromatograms and mass spectra (blanks, standards & 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 LIMS-plus. The ASCL case number, exhibit 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.

B. Positive Results

Any compound(s) meeting the criteria for positive results, as defined in section 5.4.5.6. part 5.B., 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.²³

C. Indicative Results

Any compound(s) meeting the criteria for indicative results, as defined in section 5.4.5.6. Part 5.C., 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.²⁴

D. Negative Results

- i. Any compound(s) meeting the criteria for negative results, as defined in section 5.4.5.6. part 5.D., are generally not included in the notes.

²³ Known breakdown products and manufacturing byproducts that are controlled are excluded from this requirement.

²⁴ Ibid

- ii. If the chromatogram contains no peaks, or if the mass spectra of all peaks in the sample's chromatogram do not visually match any available reference standard spectra, then the results may be recorded in the case notes as e.g. "No peaks", "No match", "No ID" etc. (If the chemist desires to list the best software algorithm match(s) of the mass spectra of peaks in the sample to available library reference spectra, the match(s) should be enclosed in brackets.)

This copy is not controlled.

5.4.5.7. Energy Dispersive X-Ray Fluorescence (EDXRF)

Scope: The methods in this document describe the techniques used to prepare and obtain elemental spectra from solid and liquid phase samples, precautions and possible sources of error, data interpretation and notations specific to this test. Arrangements to run samples on the XRF instrument in Little Rock should be made through the appropriate Supervisor.

1. Calibration & Maintenance

Refer to section 5.5.6.

2. Instrument Operation Parameters

Table 5.4.5.7.-1 Routine Instrument Parameters for X-ray Fluorescence	
X-Ray Path	Vacuum
Voltage	15 kV
Counts per Second (CPS) ¹	2000-5000
Dead Time (DT%) ¹	25%
* These should be considered starting point values only and may be adjusted by the Forensic Chemist depending on the type of information needed. ¹ Both CPS and DT% are functions of current and therefore are adjusted by raising and lowering the current.	

3. Technique(s)

A. Sample Preparation

- i. A piece of disposable thin film (e.g. Mylar® or Ultralene®) is secured over one end of the plastic cup holder (labeled with the last five digits of the full laboratory case number and item number) with a plastic cup holder ring.
- ii. Sample is transferred to the holder...
 - a. **If the sample is a solid:** The solid is placed inside the plastic cup holder on the disposable thin film. A piece of wiping paper or filter paper is used to fill the plastic cup holder.
 - b. **If the sample is a liquid:** The sample is spotted onto a piece of wiping paper or filter paper and allowed to dry.
[NOTE: If the sample is suspected to be crystalline iodine, a suitable solvent must be added to the suspected iodine crystals and the solution spotted onto a piece of wiping paper or filter paper and allowed to dry.]
- iii. A second piece of disposable thin film is placed of the other end of the plastic cup holder, secured with a plastic cup holder ring, and a very small hole(s) punched into the top piece of disposable thin film.

B. Running the Sample(s)

The plastic cup is placed into the instrument's sample holder and a spectrum is acquired. It may be necessary to repeat these steps and adjust operating parameters (adjusting μA will effect CPS, dead time, and spectrum intensity; kV

must be at least 5kV above the energy of the element of interest and high enough to excite the entire sample) to obtain the best possible results.

4. Precautions & Possible Sources of Error

- Elements below sodium on the periodic table cannot be detected. Nitrogen, oxygen, fluorine, and neon can only be seen under special conditions and through special preparations.
- The oxidation state of the elements detected cannot be determined.
- Contamination of the sample compartment.
- Contamination of sample holder and/or rings (i.e. iodine stains).
- Sample placement in sample holder: Best results are obtained when as much of the sample as possible is in the middle of the cup rather than to one side.

5. Data Interpretation

A. General

Identification of components in a sample is based on comparing the emission lines in the sample's XRF spectrum with known XRF emission energies of the elements. 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 suggested element is actually present in the sample.

B. Positive Results

Line(s) in the sample's spectrum visually matches that of the reference standard and the following criteria are met:

- i. All major emission lines for the element are present (i.e. proper energies and patterns) in the sample.
- ii. All emission line intensities in the sample are in the proper ratios compared to the element. (Exception: If there is overlap between emission lines of more than one element, these line(s) may be omitted from ratio comparison. Both overlapping lines should be labeled.)
- iii. The sample must have a net intensity of 0.100 cps/ μ A for the element of interest.

C. Indicative Results

N/A

D. Negative Results

Peak(s) are not positively identified, or there are no peaks present.

6. Notations Specific to This Test

A. General

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

i. Case Notes

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

- a. Type of sample preparation(s) (if applicable), and
- b. the date of the testing.

ii. Instrumental 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 LIMS-plus. The ASCL case number, exhibit number, and date must be visible on the image.

B. Positive Results

Any element(s) meeting the criteria for positive results, as defined in section 5.4.5.7 part 5B, will be entered into the case notes by the element name or an appropriate abbreviation.

C. Indicative Results

N/A

D. Negative Results

Samples meeting the criteria for negative results, as defined in section 5.4.5.7 part 5D, will be entered into the case notes by e.g. "No match," "Nothing significant" or "Negative (-)."

5.4.5.8. *Pharmaceutical Identifier*

Scope: The methods described in this document can be used to aid in the identification of pharmaceuticals, in the form of tablets and capsules²⁵, submitted for drug analysis.

1. **General Method**

- Record the physical appearance of the tablet/capsule (e.g. imprint, color, shape, scoring, etc.) in the case notes.
- Compare sample characteristics/appearance to reference source(s).

2. **Reference Sources for identification**

- DIB (any year)
- IdentaDrug (any year)
- ASCL-Logo ID (spreadsheet)
- Poison Control
- Drugs.com (pill identifier only)
- pillbox.nlm.nih.gov
- manufacturer sources of information (conversations, e-mails, and manufacturer produced ID books)

3. **Precautions & Possible Sources of Error**

- Pharmaceuticals containing similar imprint information.
- Counterfeit items.
- Imprints matching multiple identifications.

4. **Data Interpretation**

A. General

Pharmaceutical references are used to presumptively identify commercial pharmaceutical products.

B. Positive Results

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

C. Indicative Results

²⁵ Sealed, commercially available pharmaceutical items in other forms (e.g. ampules, patches, etc.), without any apparent tampering can be positively identified for reporting based on the manufacturer's labeling.

Broken or partial tablets or damaged capsules largely match the description from a reference source but some markings are not visible.

D. 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.

5. Notations Specification to This Test

A. General

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

B. Positive Results

The active ingredient(s) of any tablet/capsule identifications meeting the criteria for positive results, as defined in section 5.4.5.8. part 4B., will be entered into the case notes by name or an appropriate abbreviation. The dosage, manufacturer, and the reference source(s) used to make the identification will also be documented in the case notes.

C. Indicative Results

The possible active ingredient(s) of any tablet/capsule identifications meeting the criteria for indicative results, as defined in section 5.4.5.8. part 4C., 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, manufacturer, and the reference source(s) used to make the identification will also be documented in the case notes.

D. Negative Results

The case notes will indicate that no identification could be made for tablets/capsules meeting the criteria for negative results, as defined in section 5.4.5.8. part 4D.

5.4.6. Estimation of Uncertainty of Measurement

1. General

A measurement of uncertainty for the analytical balance, toploader balance, bulk balance, and quantitation of methamphetamine will be maintained by the laboratory.

2. Procedures

An explanation of the procedure can be found on S:\SOP clarifications\MOU documents\measurement of uncertainty explanation.

Recalculation of the measurement of uncertainty estimates shall occur when there are significant changes to the procedures or environment; or when there are personnel changes to the section.

An explanation on MoU balance measurements and reporting can be found in section 5.4.4.1.A & B. of this manual.

An explanation on MoU quantitation reporting can be found in section 5.10.1.C.v. of this manual.

3. Sources of Uncertainty

The measurement of uncertainty budgets can be found on S:\SOP clarifications\MOU documents\MOU Budget Form.

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5.5. Equipment

5.5.1. General

The ASCL has adequate instrumentation and equipment to perform the necessary testing.

1. Training

Only individuals who have been trained in the proper use of the instrumentation or equipment are authorized to use it. The training program for new employees includes familiarization with all instrumentation (hardware and software) and equipment associated with the testing techniques described in this manual and available at their work site. When new instrumentation or equipment is acquired and requires a validation, appropriate personnel will be trained, and this training will be documented and kept in each individual's Employee History Binder. Up-to-date instructions on the use and maintenance of the instrument or equipment will be readily available for use.

2. Identification

All instruments and equipment will be uniquely identified with a serial number or asset number. Each instrument used in data analysis will be assigned a unique identifier.

3. Records

All calibrations, performance verifications, and maintenance will be properly documented in a log. This log will be located near the instrument and readily available to each analyst who uses the instrument or equipment.

When instrumentation or equipment is retired, the associated records shall be maintained and available for at least one full accreditation cycle (4 years).

4. Handling and Maintenance

A. General

The maintenance of equipment and instrumentation having a significant impact on the quality of results is a planned activity. Primary responsibility for this maintenance rests on the personnel in the discipline. Major classes of instrumentation and equipment employed by the discipline are treated individually in the following subsections of 5.5. These subsections describe performance verification checks and preventive maintenance (if applicable). All instrumentation and equipment will be maintained in a clean, orderly, and safe

condition. Laboratory equipment and instrumentation will be handled responsibly to ensure optimal performance and to avoid contamination and premature wear and damage.

It is the responsibility of the appropriate supervisor to ensure that proper planning and care is taken when equipment or instrumentation is initially located or subsequently moved. Due care shall be taken if equipment or instrumentation is to be shipped to a manufacturer or vendor for calibration or maintenance to minimize the possibility of damage in transit. Equipment that is infrequently used will be stored (covered, powered-down, etc.) per the manufacturer's recommendations.

B. Intermediate Performance Verifications

Refer to subsections below for specific requirements.

C. Adjustments

After a performance verification has been performed, it may be necessary to make adjustments to the instrument or equipment utilizing certified or traceable reference standards/materials (e.g. balance, GCMS). These adjustments shall be documented in the calibration/performance verification log.

D. 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 log 'OUT OF SERVICE' in order to prevent inadvertent use of the equipment before they seek help in resolving the problem. Any problem and the action to correct the problem must be recorded in the instrument or equipment's log. When it has been determined that instrumentation or equipment was not working properly, the appropriate supervisor shall take into consideration the effect the problem may have had on previous tests (see Section 4.9). Instrumentation or equipment taken out of service will not be used in casework until appropriate calibration or performance verification is performed.

Newly acquired instruments that do not have performance verification procedures are 'OUT OF SERVICE' and may not be used for casework until those procedures are determined by the appropriate supervisor, any necessary

revisions are made to this manual, and the instrument passes its performance verification.

E. Protection and Security

Instrumentation or equipment with calibration settings that can be adjusted by laboratory personnel will be safeguarded against unintentional changes which would invalidate the test results. This may be accomplished by one the following:

- Utilizing positive/negative controls, standards or known reference material at the beginning and end of instrumental runs/analytical sequences;
- Tamper proof seals placed over the adjustment points;
- Dedicated personnel as the only individuals authorized to make the adjustments.

All GCMS and GC methods are password protected. Supervisors will disseminate those passwords to additional personnel on an as-needed basis.

F. Calibration Status

Balances are the only piece of equipment that will require calibration. Balances will be calibrated every five (5) years by an outside vendor designated by the appropriate supervisor (see also section 5.6). The records of the calibrations will be retained by the supervisor.

G. Outside Maintenance

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.

5.5.2. Balances

Performance Verification & Maintenance (Preventive)

An *Analytical* or *Toploading Balance Performance Verification Log Sheet* is provided to each chemist for each balance they are issued for use. Each chemist's balance(s) will be subjected to the performance checks in TABLE 5.5.2.-1 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. Log sheets are filed at the end of each month and archived.

TABLE 5.5.2.-1 Routine Daily Balance Checks	
Daily Checks	Actions
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 adjustments before use ☹ If yes, Balance ready to use ☺
* Analytical balance – 100 g ± 0.5 mg, Toploader balance – 100 g ± 0.00g, Bulk balance (bulk room) – 2000 g ± 0.00 g, Bulk balance (evidence) – 10 kg ± 0.00 kg	

In addition to the daily performance checks, the chemist will subject their balance(s) to the performance checks in TABLE 5.5.2.-2 on a monthly basis. The results of these checks and the serial number (or identifying number) of the calibrated weight used for the checks will also be recorded on the appropriate log sheet.

TABLE 5.5.2.-2 Required Monthly Balance Checks	
Monthly Checks	Actions
Has the balance been performance checked with two other weights (e.g. 1g and 20 g for top-loading balance)?	Weigh and record the weights
Was the balance within tolerance?*	If no, perform adjustments before use ☹ If yes, Balance is ready to use ☺
* Analytical balance – 1 g ± 0.2 mg, Analytical balance – 200 mg ± 0.1 mg; Toploading balance – 20 g ± 0.00 g, 2000 g ± 0.00 g; Bulk balance (bulk room - LR) – 4000 g ± 0.00 g, 25 lb. ± 0.00 lb.; Ohaus ES50L balance – 20 kg ± 0.00 kg, 50 kg ± 0.00 kg	

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. 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 the chemist must use a balance other than their personal issue (e.g. another chemist's balance or the bulk scale), 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 should indicate in their notes which weighing(s) were done on a balance they do not ordinarily use, and which balance was used.

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5.5.3. Infrared Instruments

1. Performance Verification

The performance verification of each FTIR must be checked monthly²⁶ 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 Hope), and software version. The FTIR bench and ATR accessory are separate entities 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.

A. Nicolet 380 FTIR with ATR Accessory

- i. Remove the purged air connection from the instrument.
- ii. Remove the ATR accessory.
- iii. Place transmission plate in the FTIR (a screen should appear indicating 'transmission experiment setup') and place the FTIR cover on instrument.
- iv. Align the bench (>Collect>Experiment Setup>Diagnostic>Align).
- v. Run the Valpro Qualification (>Analyze>Valpro Qualification>Nicolet 380 System KBr-EP).
- vi. A ValPro Qualification Report will appear on the screen. If all tests have passed, index the qualification report to the designated ASCL case # for the instrument. Document the results of tests on the *FTIR-ATR Logsheet*.
- vii. Remove the FTIR cover and transmission plate and place the ATR accessory back on the instrument (a screen should pop up indicating a 'smart accessory change'; click 'OK'. A 'Test Smart Accessory' screen should appear. Once it says 'all tests passed', click 'OK'.).
- viii. Run the Valpro Qualification (Analyze>Valpro Qualification>Smart Orbit Diamond Accessory-EP). Place the 'Lolipop' polystyrene standard on the diamond crystal. Use the anvil to tighten down into position forcing the standard against the crystal.
- ix. A ValPro Qualification Report will appear on the screen. If all tests have passed, index the qualification report to the designated ASCL case # for the instrument. Document the results of tests on the *FTIR-ATR Logsheet*.
- x. Plug the purged air back into the instrument. Allow approximately 10 minutes for the air to equilibrate before running the background and using in casework.

²⁶ Months in which an instrument is not used in casework are excepted from this requirement.

- xi. If the instrument failed any of these tests, the instrument must be removed from service for repair.

B. Nicolet Avatar

- i. Begin with the transmission accessory removed and the sample compartment cover in place. Scan a new background.
- ii. Place polystyrene sample card into the sample compartment.
- iii. Run the macro POLYQC.MAC.
- iv. Assess the report for the pass/fail requirements.
- v. If the instrument passes all checks then the bench is ready to use for transmission/absorption experiments. Incorporate an image of the report into the current year's electronic logbook for the instrument and record the results on the FTIR Log Sheet. If the instrument fails any of these checks then remove it from service for repair.

Complete the following steps if ATR experiments are to be conducted.

- vi. Insert the ATR smart accessory. Allow the instrument to detect and test the smart accessory. Verify that the software shows all tests passed (green check in the dialog window or the Bench tab in Experimental Setup shows that the interferogram peak amplitude is "within acceptable range") for the accessory.
- vii. If the ATR passes all checks then note the results on the FTIR Log Sheet. If the ATR fails its checks then remove it from service and consult the supervisor.

C. Nicolet iS10

- i. Begin with the transmission accessory removed and the sample compartment cover in place.
- ii. Run the Performance Verification report.
- iii. Assess the report and polystyrene standard spectrum for the pass/fail requirements.
- iv. If the instrument passes all checks then the bench is ready to use for transmission/absorption experiments. Incorporate an image of the report into the current year's electronic logbook for the instrument and record the results on the FTIR Log Sheet. If the instrument fails any of these checks then remove it from service for repair.

Complete the following steps if ATR experiments are to be conducted.

- v. Insert the ATR smart accessory. Allow the instrument to detect and test the smart accessory. Verify that the software shows all tests passed (green check

- in the dialog window or the Bench tab in Experimental Setup shows that the interferogram peak amplitude is “within acceptable range”) for the accessory.
- vi. If the ATR passes all checks then note the results on the FTIR Log Sheet. If the ATR fails its checks then remove it from service and consult the supervisor.

2. Maintenance

Maintenance is performed on an as needed basis. All maintenance must be documented on the *FTIR Maintenance Log Sheet*. The performance verification listed above must be performed before returning the instrument to service.

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5.5.4. Gas Chromatographs

Performance Verification & Maintenance (Preventive)

A *GC Log Sheet* will be provided for each instrument. This log sheet will be used by each chemist using the instrument to track the number of injections, when septa and injection liners were changed, when check samples were run, and when a check sample using a new batch of internal standard was first run.

1. Routine Instrument Maintenance

TABLE 5.5.4.-1 Daily Routine Maintenance Checks	
Daily Checks	Action
Number of injections	If >~150, replace septum and injection liner
Solvent wash vials	Empty, rinse, and refill with methanol
Waste vials	Empty and rinse with methanol

2. Calibration Curve Maintenance

TABLE 5.5.4.-2 Daily Calibration Curve Checks	
Daily Checks	Action
Has a check sample been run for the substance to be quantitated?	If yes, proceed to next step ☺ If no, run a solvent blank and the check sample ☹
Did the calibration curve identify the substance to be quantitated and the internal standard?	If yes, proceed to the next step ☺ If no, contact the Section Chief or designee ☹
Was the percent error $\leq 5\%$?	If yes, initial the printout and the calibration curve is ready for use ☺ If no, calibration curve is removed from service until error is resolved ☹

3. Non-Routine Maintenance

A *GC Maintenance Log Sheet* will be provided for each instrument. The chemist performing maintenance or repairs will document these on the maintenance log sheet. Non-routine maintenance is performed on an as needed basis.

5.5.5. Gas Chromatograph/Mass Spectrometers

Purpose: To provide a guideline for the proper care and maintenance for the GC/MS instruments in the Forensic Chemistry Section.

1. Performance Verification

A. Daily Performance Verification

Each GC/MS should be performance checked before use, using the Autotune feature of the Chemstation software. The 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).

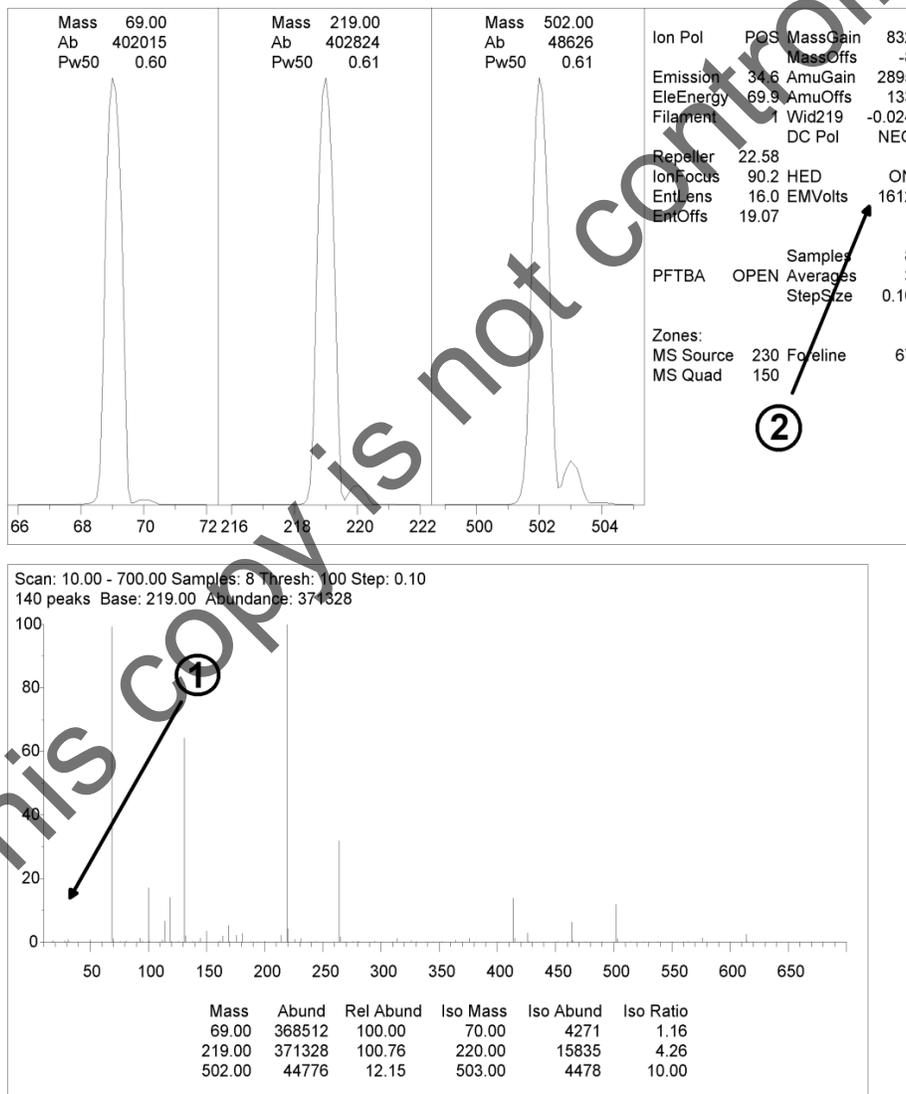


FIG. 1 Important Areas of the MSD Report.

The chemist must assess the performance check by examining the labeled areas of the report (FIG. 1①,②) for the following conditions:

- ① Abundance of any peak(s) below 69 m/z (e.g. 18[water], 28[nitrogen], 32[oxygen]) are >20%, relative to the abundance of the peak at mass 69,
- ② and EM voltage > 2900.

If either of these conditions exists, the instrument is not in proper working condition and should be removed from service until it has been repaired and has passed a performance check.

All reports will be indexed to the designated ASCL case # for the instrument.

B. New Instrument Performance Verification

After the instrument manufacturer installation is complete and the Forensic Chemistry section been given approval from the manufacturer to place the instrument in use, the following procedure shall be performed:

1. Copy all routine methods from an instrument with compatible operating software.
2. Run each routine method with either a test mix of drug standards that are commonly identified using that method that includes a drug that elutes early in the temperature ramp and one that elutes late in the temperature ramp, or a single drug that is commonly identified with that method.
3. Evaluate the chromatography, fragmentation patterns, and library matching capabilities to ensure quality performance of the instrument and software.
4. Retain this information in the appropriate location.

C. New Method Performance Verification

When a new method is designed for routine use, a performance verification shall be performed to ensure quality performance before it is put into use.

1. Run the new method with either a test mix of drug standards that are commonly identified using that method that includes a drug that elutes early in the temperature ramp and one that elutes late in the temperature ramp, or a single drug that is commonly identified with that method.
2. Evaluate the chromatography, fragmentation patterns, and library matching capabilities to ensure quality performance of the instrument and software.
3. Retain this information in the appropriate location.

4. Copy the newly designed method to all other compatible instruments currently in service.

2. Routine Maintenance (Preventive)

A *GC/MS Log Sheet* will be provided for each instrument. This log sheet will be used by each chemist using the instrument to track the number of injections, when septa and injection liners were changed, when performance checks were performed and when filaments have blown.

TABLE 5.5.5.-1 Daily Routine Maintenance Checks	
Daily Checks	Action
Number of injections	If > ~100, replace septum and injection liner
Solvent wash vials	Empty, rinse and refill with methanol
Waste vials	Empty and rinse with methanol
Mass Spectrometer not calibrated	Autotune the Mass Spectrometer

3. Non-routine Maintenance

A *GC/MS Maintenance Log Sheet* will be provided for each instrument. The chemist performing maintenance or repairs will complete the maintenance log sheet with repairs to the instrument noted in the comment field. After maintenance or repairs are completed the chemist shall perform a performance check the instrument before it is returned to service.

Other maintenance is performed on an as needed basis. When the GC/MS has been removed from service for this maintenance, the following checks and actions should be performed on the following items:

TABLE 5.5.5.-2 Non-Routine Maintenance Checks	
Serviceable Part	Action
Source	Clean according to Agilent procedures
Filaments	Replace
Diffusion pump oil	Inspect and fill or replace if necessary
Fore-line pump oil	Check and fill or replace if necessary
Vent line	Rinse with methanol
Vent-line trap	Inspect and replace if necessary
Gold inlet seal	Inspect and replace if necessary
PFTBA level	Check and fill if necessary

5.5.6. Energy Dispersive X-Ray Fluorometers

1. Performance Verification

A verification sample should be run daily before use to insure that the instrument is in proper working condition, using a stainless steel disk. The specifications for passing verification are the following:

Fe	65.5 – 70.0%	Energy for Fe: 6.40 ± 0.05 keV
Cr	14.75 – 20.0%	Resolution for Fe: <170eV
Ni	8.75 – 13.25%	
Mn	0.5 – 3.5%	

The verification report will be indexed to the designated ASCL case # for the instrument. The results of the verification sample should be documented on the XRF Logsheet in the *XRF Logbook*.

2. Routine Maintenance (Preventive)

This instrument requires the detector to be cooled by liquid nitrogen. The Dewar for this instrument should be filled as needed. This is documented in the *XRF Logbook*.

The sample compartment is cleaned on an as needed basis and documented in the logbook.

3. Non-Routine Maintenance

All other maintenance should be documented in the *XRF Logbook*. When the XRF will not pass verification samples, it will be necessary to perform an adjustment using a disk composed of aluminum and copper, and the EDX software (under "instrument calibration").

5.6. Measurement Traceability

1. Scope

This section will address measure traceability and the documented procedures for the calibration and performance checks of measurement equipment and for the reference standards used to demonstrate traceability.

2. Calibration Requirements

Balances will be calibrated every five (5) years. The calibration records will be retained by the appropriate supervisor. All balances and FID gas chromatographs are considered category 1 equipment. All other equipment in the drug chemistry discipline is considered category 2.

3. Performance Verification

See Section 5.5.

4. Intermediate Checks

See Section 5.5.

5. Testing

A. Unit Traceability and External Calibration Services

The calibration or performance check of the instrumentation/equipment, whenever possible, will be traceable to the International System of Units (SI). When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability, and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty or a statement of compliance with an identified metrological specification.

B. Non-SI Unit Traceability

There are certain comparisons, in the Drug Chemistry Discipline, that currently cannot be strictly made in SI units. They are as follows:

- Drug Standards used to check the calibration curve on the GC
- PFTBA that is used to autotune the GCMS
- Polystyrene used to performance check the ATR
- The standards used to performance check the XRF

In these cases, verification provides confidence in measurement by establishing traceability to appropriate measurement standards through the use of

- certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material
- specified methods, or consensus standards that are clearly described and agreed by all parties concerned

6. Reference Standards and Reference Materials

A. Reference Standards

Reference standards shall be calibrated by a body that can provide competence, measurement capability, and traceability. Such reference standards of measurement held by the laboratory shall be used for performance verifications or adjustments only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

NIST certified weights are used to conduct performance verifications and adjust the balances used for casework. The weights will be calibrated or replaced every ten (10) years. The calibration records will be retained by the appropriate supervisor.

B. Reference Materials

All reference materials, whether prepared in-house or purchased from commercial sources, must be verified prior to use. A Certificate of Analysis will suffice for verification. Materials for which no certificate of analysis is provided must be verified before use through infra-red spectroscopy or mass spectrometry.

Substances encountered in casework may be collected for use as reference materials. These substances, referred to as secondary standards, are verified during case analysis, and may be used for comparison purposes in subsequent casework. [The supervisor must inspect the substance for suitability as a reference material and approve its collection.] The submitting agency must be notified of the amount taken for this purpose. Secondary standards must be stored in the locked drug cabinet and are subject to the reference collection requirements below.

C. Reference Collections

Reference collections of data (e.g. mass spectral or other libraries), or items/materials encountered in casework which are maintained for identification,

comparison or interpretation purposes shall be fully documented, uniquely identified and properly controlled.

7. Transportation and Storage

Reference standards/materials shall be handled responsibly to prevent contamination or deterioration and in order to protect their integrity. It is the supervisor or designee's responsibility to ensure that proper planning and care is taken.

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5.7. Sampling, Sampling Plans, and Sample Selection

The analysis of many of the cases received by the section is very straightforward. However chemists are routinely presented with more complex cases (that are poly-drug, multi-exhibit, exhibits composed of sub-items etc.) that require them to make decisions about which items or parts of items are tested. This section is meant to supplement the chemist's experience and training and give specific instructions in situations likely to arise as they process cases.

DEFINITIONS

Sampling: Taking a part of a substance, material, or product for testing in order to reach a conclusion, make an inference about, and report on the whole. Sampling should only be used when there is a reasonable assumption of homogeneity of the whole.

Sampling Plan: For an item that consists of multi-unit population (e.g., tablets, baggies, bindles), a sampling plan is a statistically valid approach to determine the number of sub-items that must be tested in order to make an inference about the whole population.

Sampling Procedure: A defined procedure used to collect a sample or samples from the larger whole, to ensure that the value obtained in the analysis is representative of the whole. The sampling procedure may include details about size and number of sample(s) to be collected, locations from which to collect the sample(s), and a method to ensure the homogeneity of the larger whole (or to make it so).

Sample Selection: A practice of selecting items to test, or portions of items to test, based on training, experience, and competence. In sample selection, there is no assumption about homogeneity.

Sample: The portion(s) of an evidence item retained, regardless of whether through sample selection or a sampling procedure, and subjected to analytical testing.

1. Sample Selection vs. Sampling

The section's overall goal is to analyze a sufficient number of items to substantiate the highest possible charge(s) for each drug schedule represented in the case while also minimizing the number of items tested. In the majority of cases, sample selection will be employed.

A sampling procedure must be followed if quantitative information is needed. Sampling may also be appropriate for large multi-item populations where it's desirable to draw a statistically valid conclusion about the presence of a drug in the whole population without having to test each item. A statistical sampling plan is provided for exhibits consisting of multi-unit populations and may only be used with prior approval of the prosecutor.

A population can consist of a single unit or multiple units. A multi-unit population has all of the following characteristics:

- Sub-items must exist in discrete forms (e.g., tablets, baggies, bindles)
- The appearance of the sub-items is essentially the same.

2. Selecting Items to Test

A. General

For cases containing multiple exhibits, the chemist working the case will select exhibits to test based on their training and experience. Items selected for testing from multi-unit populations will also normally be selected based on training and experience²⁷. Items listed as probable cause for a search should be selected for analysis. Cross contamination of items may preclude the examination of the contaminated items(s).

When three or more different scheduled drugs are present in the same case, the drugs whose schedule and amount achieves the highest charge will be tested to the maximum threshold. The remaining two or more types of scheduled drugs will be tested minimally to show presence only.

Negative 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.

B. Tablets/Capsules

The chemist will inspect all the tablets or capsules in an exhibit to ensure consistency. If the tablets or capsules can be pharmaceutically identified to contain no controlled substances then no further testing is required. If the tablets or capsules cannot be pharmaceutically identified or are identified to contain a controlled substance, then see this section 3.B.i.a.

C. Drug Paraphernalia

Chemists are not required to analyze items of paraphernalia unless:

- i. Paraphernalia is the only evidence in a case²⁸, or

²⁷ See this section 3.C.iii. to determine if sampling is appropriate instead.

²⁸ If multiple items of paraphernalia are present in the case, an item that in the chemist's opinion is most likely to test positive for the presence of methamphetamine or cocaine, and serves the purpose to inject, ingest, inhale, or otherwise introduce into the human body a controlled substance should be selected.

- ii. The item of paraphernalia serves the purpose to inject, ingest, inhale, or otherwise introduce into the human body a controlled substance and weighable amounts of cocaine or methamphetamine are present in the case along with one or more other controlled substances.

3. Collecting a Sample

A. General

The analyst must make every effort to preserve a portion of each evidence item tested.

B. Sample Selection

- i. Solids: Based on training and experience, retain a sample for analysis suitable to reach a conclusion.
 - a. Multi-unit Tablets or Capsules: A single portion of the population will be tested. Avoid collecting whole tablets or capsules. Obtain a sample composed of a sufficient number of half tablets (or approximately half the contents in the case of capsules) to reach a conclusion.
 - b. Multi-unit Solid Dosage Forms of Suspected LSD: A single portion of the multi-unit population will be tested. Obtain a sufficient number of blotter squares (or other dosage form) to reach a conclusion. The tested portion should be sealed, marked, and returned with the evidence when practical.
- ii. Paraphernalia
A sample may be obtained by rinsing items of paraphernalia with a suitable solvent.

C. Sampling

- i. Procedure for Liquids
Single layer liquids have a reasonable assumption of homogeneity. 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. Multi-layer liquids, not part of controlled substance manufacturing case, require collection of a portion of each layer²⁹.
- ii. Procedure for Single-unit Solids: Quantitative Analysis Desired
*Items to be **quantitated*** must be homogenized before sampling. Excessively wet samples should be dried prior to homogenization. This may be

²⁹ Test and report the results of each layer independently.

accomplished by allowing to air dry in a ventilation hood. The sample(s) must be clearly marked as "evidence" and should be in a secure area. Case notes should indicate weights taken both before and after drying.

Homogenize the entire exhibit to be analyzed by grinding / powdering / mixing as appropriate. Reserve a sufficient portion of the processed material as the quantitative analysis sample.

iii. Multi-item Populations

If the chemist and supervisor determine that sampling of a multi-item population will result in significantly fewer tested samples than testing to the maximum charge, the chemist will contact the prosecutor to obtain approval for sampling the population. The chemist should 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 so that the prosecutor may make an informed decision.

a. Sampling Plan

The 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 Table 5.7 -1³⁰ to determine the number of sub-items that must be independently tested.

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³⁰ Alternatively, use spreadsheet S: \Hypergeometric Calculator.xls to calculate the actual sample size required.

- b. Sampling Procedure
Randomly select³¹ from the population the number of sub-items determined by the sampling plan. For each sub-item, obtain an independent³² portion for analysis suitable to reach a conclusion. Clearly label the items that were selected to form the sample.

Table 5.7.-1 Hypergeometric Distribution			
Population Size N	Sample Size*	Population Size N	Sample Size*
5	4	30	17
6	5	40	18
7-8	6	50	19
9	7	60	20
10-11	8	70	21
12-13	9	80	22
14-15	10	90-100	23
16-17	11	101-200	26
18-20	12	300-400	27
21-23	13	500-4999	28
24-27	14	5000-10000	29
28-29	15	>10000	See S:\Hypergeometric Calculator.xls

* Required sample size to guarantee with 95% confidence that the seizure contains at least a proportion of drugs when k=0.9, if expected that all sampled units contain drugs. If a population size falls in between population sizes listed in the chart, sample for the higher population size.

4. Labeling of Sample Containers

All evidence sample containers (i.e. test tubes, beakers, auto sampler vials, etc.) must be labeled at a minimum with the last five (5) digits of the ASCL case number and the exhibit number.

³¹ e.g. number the items and use a random number generator to select the specific items to test.

³² i.e. not combined.

5.8. Handling of Test Items

1. Standard Evidence Handling

A. Chain of Custody

Transfers of evidence are tracked within and between the Little Rock and Hope laboratories using JusticeTrax LIMS-plus. At the Hope laboratory, evidence is typically transferred from the custody of Evidence Receiving to the analyst assigned to process the evidence and is returned by the analyst to Evidence Receiving after all necessary analyses are performed. At the Little Rock laboratory, evidence is typically transferred from Evidence Receiving to FCSecureStorage and is returned to FCSecureStorage and back to Evidence Receiving after all analyses are done. The FCSecureStorage area is only available to the members of the Forensic Drug Chemistry Section and other authorized personnel. Documented evidence transfers between analysts may occur, as deemed necessary.

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

The supervisor 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.

B. Suitability of Test Items

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, the analyst's supervisor and the customer agency shall be advised and consulted for further instructions.

If the analyst discovers a significant inconsistency between the stated and actual contents of a package or the suitability of an evidence item for testing, the analyst shall make an attempt to contact the customer and document the discussion (*Agency Contact Form* (ASCL-FORM-06, email, etc.) prior to issuing a report.

All remedial actions taken to correct packaging or evidence deficiencies shall be noted in the case record (e.g. submission form or analyst's notes).

C. Evidence Sealing

When an evidence container is opened, the original seal shall be left intact, whenever practical, and a new opening made.

Upon completion of sampling, the evidence or its proximal container should be sealed and marked for identification to preserve the condition and integrity of the evidence. Evidence will be sealed in a manner in which the contents cannot readily escape and opening the container would result in obvious damage or alteration to the container or its seal. Evidence (or its proximal container) will be marked with the unique ASCL case number (YYYY-00000), item number, and the chemist's initials. When heat-sealing, the analyst's initials will be made across each heat-seal.

When the analysis or examination is completed, the new opening shall be sealed, as outlined in these procedures; thus the original container seals will be intact and all seals will be clearly marked. Chemists should place their initials across their seal on the outermost packaging.

If reusing the original container is impractical, a new evidence container may be used. It shall also be marked and sealed according to the above procedures and the original evidence packaging shall be kept inside the second evidence container. If the original packaging cannot be kept, there must be complete documentation along with a picture of original packaging retained in the case record. Documentation of the change in packaging along with description must be documented in the case record for future reference.

D. Test Item Identification

If testing requires that uniquely identified items be subdivided within the laboratory, appropriate sub-item identifiers shall be assigned and the item(s) labeled by the analyst so that the sub-item may be easily tracked and identified as having originated from a particular item.

E. Safeguarding the Integrity of Evidence

It is the responsibility of the analyst to maintain proper control of all evidence in their possession. During the sampling of evidence, it may be necessary to have unsealed evidence in the analysis area (a limited access area). This time should be kept to a minimum and resealing of evidence should be performed as soon as practical. Evidence, including sampled items, should be secured in personal storage areas during lunch hours and when leaving the analysis area for any extended period of time. 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.

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Little Rock:

- The south elevator or stairway (when available) will be used to transport evidence to and from the Evidence Receiving Section. Each chemist will personally receive evidence from FCSecureStorage or an Evidence Receiving Technician (or authorized individual) and is responsible for making sure the correct evidence is received. All evidence will be processed in limited access areas. Upon completion of sampling, the analyst will personally return the evidence to FCSecureStorage or the Evidence Receiving Section. The section chief may set limits on the number of cases checked out or retained in an analyst's storage area(s).
- When an analyst needs additional storage, there is a common lockable storage cabinet(s) with an assignable key and logsheet (only one analyst will have access to this cabinet at a time).

F. Unattended Evidence

Evidence in the process of examination may be left unattended for limited periods of time (e.g. short breaks) but must be in a secure limited access area. If the analyst needs to be away for a longer period of time, the evidence shall be secured in a short term storage location, whenever practical. If this is not possible, the analyst shall take reasonable precautions to protect the evidence from loss, cross-transfer, contamination/or deleterious change.

Evidence shall not be left unattended if it is not in the process of being examined or there is no expectation of frequent examination.

G. Evidence in the Process of Examination

Items with an expectation of frequent analysis may be considered "evidence in the process of examination/analysis" and may be stored unsealed in a limited access area as long as the evidence is protected from loss, cross-transfer, contamination and/or deleterious change. After 60 consecutive days of no analysis or new requests for comparisons, a case is no longer considered "in the process of examination." Cases no longer in the process of examination should be closed and the evidence sealed properly until analysis resumes or a new service request is received.

2. Crime Scene Evidence

A. Responsibilities and Procedures

The following policies and procedures apply exclusively to the Little Rock laboratory. The Hope laboratory does not accept evidence associated with controlled substance manufacturing cases.

Evidence collected from a crime scene by ASCL personnel shall be protected from loss, cross transfer, contamination, and/or deleterious change, whether in a sealed or unsealed container, during transportation to the ASCL. Where appropriate, further processing to preserve, evaluate, document, or render evidence safe shall be accomplished prior to final packaging. The evidence shall be appropriately identified, packaged, and entered into JusticeTrax LIMS-plus as soon as practical.

All externally submitted illicit lab evidence is to be inspected by a forensic chemist, or other employee with the appropriate chemistry background and training. The *Illicit Laboratory Safety Form* (ER-FORM-01) will be utilized and signed by a chemist certifying that the evidence has been checked. The evidence inspection procedure is located in this section under Crime Scene Evidence.

In order to determine the items most likely to assist in the investigation and prioritize those items for examination, the examiner or analyst may conduct a review of large, bulky submissions. Whenever possible, this review will occur with the agency representative in person or by phone to assist with the investigation and to eliminate unnecessary examinations or analyses.

B. Submission of Evidence

A wide range of chemical evidence could be submitted by an officer. Some of the evidence submitted may pertain to specific charges of manufacturing a controlled substance or some of the lesser charges similar to manufacturing. Some of the samples in these types of submissions may contain organic powders, inorganic powders, organic solvents, strong aqueous bases, strong aqueous acids, pyroforic metals, noxious gases, or flammable vapors. Any time an evidence technician is aware of charges pertaining to manufacturing or its lesser charges, a member of the illicit laboratory team should be called to render the samples safe for storage. In the event that an Illicit Laboratory section member is not present, a Forensic Chemist or other employee with the appropriate chemistry background and training should be called. The chemist responsible for rendering samples safe has the authority to refuse any samples that are deemed unsafe for storage.

i. Submission of Clandestine Laboratory Evidence by Submitting Officer

- Chemical evidence consists of evidence that is recovered from suspects or crime scenes, which requires analysis by the Illicit Laboratory Chemist to help answer case questions from the investigator. After samples have been rendered safe, all packages containing evidence should be sealed and initialed across the seal by the submitting officer. The type of packaging used should prevent the loss, deterioration, or cross contamination of the evidence. The evidence list and submission sheet should be complete including all evidence items contained in the sealed package. The submission sheet should have all suspects' names including dates of birth whenever possible, agency case number, agency mailing address, date of offense, and type of offense.
- ii. Submission of Clandestine Laboratory Evidence by Illicit Laboratory Chemist
All procedures for submitting clandestine laboratory evidence by an officer should be followed when a forensic chemist is the submitting officer. All submission forms should be filled out completely. The location, submitting chemist, type of clandestine lab, and crime lab case number should be documented. Any case submitted by an Illicit Laboratory Chemist should be analyzed by the same chemist.

C. Packaging of Evidence

- i. Packaging of Volatile Chemicals
Whenever possible, all illicit laboratory evidence submitted by an officer should be inspected by a clandestine laboratory certified chemist and rendered safe. In the event that an Illicit Laboratory section member is not present, a Forensic Chemist or other employee with the appropriate chemistry background and training should be responsible for the inspection. It may be necessary for the inspecting chemist to take a representative sample of some evidence items. All remaining chemicals not needed will be returned to the submitting officer, whenever practical. 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.
- ii. 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.
- iii. Packaging of Iodine

Iodine should be packaged with great care to prevent cross contamination. Because of the sublimation properties of iodine, only a small amount (i.e. 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.

iv. Packaging of Lithium or Sodium Metal

Lithium and sodium metal are pyroforic 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 (i.e. one inch square) should be placed in a glass vial with a Teflon seal. A heavy organic solvent or petroleum distillate should be poured into the glass vial. It is important to keep the surface of the solvent well above the lithium or sodium metal.

v. Packaging of Anhydrous Ammonia

Anhydrous ammonia is a very dangerous basic gas. No anhydrous ammonia containers should 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.

vi. Packaging of Sharps

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

vii. Packaging of Biohazard Materials

Any evidence that is believed to be contaminated with biohazard material should be packaged in an OSHA approved biohazard materials container. No evidence should be submitted that is believed to be contaminated with airborne transmitted bacteria or viral material.

D. Submission Sheet Inventory

It is the responsibility of the submitting officer to insure that the submission sheet reflects the evidence items submitted; however, should the chemist involved with rendering the lab safe notice a discrepancy, the submitting officer must remedy the situation before the evidence can be submitted into the evidence receiving section of the Arkansas State Crime Laboratory.

E. Completion of Rendering Safe

Once the chemist is finished rendering the submitted samples safe, an evidence safety sheet should be filled out and placed in the Arkansas State Crime Laboratory electronic case file or equivalent. An illicit laboratory case does not have to be assigned to the chemist responsible for rendering it safe. When an illicit laboratory chemist is submitting samples obtained in the field by the submitting illicit laboratory chemist, tendering an illicit laboratory safety sheet is recommended but not required in order to submit the evidence.

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5.9. Assuring the Quality of Test Results

1. General

The Drug Chemistry Discipline maintains quality control procedures to continually monitor and ensure the validity of test results. Quality control data will be recorded in a way to allow trends to be detected and whenever practical, statistical techniques will be used to review the data. When quality control data is found to be outside the acceptable criteria, planned action shall be taken to correct the problem and to prevent incorrect results to be reported. The records will be retained to show that all appropriate quality control measures have been taken and are acceptable. The following is a list of quality control items that are utilized to ensure that test results are of the highest quality.

- regular use of verified reference materials
- where appropriate, the use of positive and negative controls and internal standards
- 100% technical and administrative review of case records prior to issuance of the laboratory report
- competency testing of analysts prior to beginning casework
- annual proficiency testing of all
- replicate testing using the same or different methods, where practical
- re-analysis of casework

The use of appropriate controls and standards shall be specified in the methods (section 5.4) and their use recorded in the case record.

2. Proficiency Testing

A. General

The Drug Chemistry Discipline maintains a proficiency testing program designed to provide independent evaluation of individual technical expertise, as well as a mechanism to monitor training needs and procedural weaknesses for both individual analysts and the section. Successfully completing a proficiency test means either obtaining the correct response or completing corrective actions pursuant to ASCL policy and/or directives from an ASCLD/LAB Proficiency Review Committee (PRC). Test specimens may be obtained from external providers or prepared internally. Internal proficiency tests may include previous external proficiency samples, samples retained from casework (secondary proficiency standards), samples prepared from primary standards, re-examination techniques, and blind techniques.

B. Participation Requirements

- i. A minimum of one chemist per laboratory participating in an external proficiency test annually from an ASCLD/LAB approved test provider.
- ii. Each chemist that does not participate in an external proficiency test must complete at least one internal proficiency test annually.
- iii. Each analyst engaged in testing activities shall be proficiency tested at least once during each four-year accreditation cycle, in each category of testing appearing on the *ASCL's Scope of Accreditation*, in which the individual performs testing.

Discipline	Categories of Testing
Drug Chemistry	Controlled Substances Quantitative Analysis General Chemical Testing (the identification and reporting of non-controlled substances) Clandestine Laboratory Analysis

C. Procedures

All internal and external proficiency tests will be assigned a case number in JusticeTrax LIMS-plus. All administration and examination records will be stored in the electronic case file. The electronic version is considered the official proficiency case record. In addition, the following will be maintained in the case file:

- How the samples were obtained or created (after testing is complete and results have been reviewed)
- Proficiency test results from provider
- *Corrective Action Request Form* (ASCL-FORM-08), when applicable

Technical and administrative review must take place just as it would with normal casework. All parts of a proficiency test provided by an approved test provider must be examined as completely as the discipline's procedures allow.

D. Evaluating Results

The appropriate supervisor is responsible for comparing the analytical results to the expected results, determining if the analytical results are acceptable, and for reviewing these results with the analyst.

The Forensic Chemistry Section uses the following criteria to evaluate the results of a proficiency test.

1. Controlled Substance/General Chemical

<i>Qualitative Analysis</i>		
	Pass	Fail
Controlled substance(s)	Correct identification of all controlled substances present.	Incorrect or incomplete identification.
General Chemical(s)	Correct identification of all general chemicals present.	Incorrect identification.

2. Quantitative Analysis

<i>Quantitative Analysis</i>		
	Pass	Fail
Controlled substance(s)	Results \leq 5% relative difference	Results $>$ 5% relative difference
Non-Controlled(s)	N/A	N/A

3. Clandestine Laboratory Analysis

<i>Qualitative Analysis</i>		
	Pass	Fail
Controlled substance(s)	Correct identification of all controlled substances present.	Incorrect or incomplete identification.
Non-Controlled substance(s)/Element(s)	Correct identification of all non-controlled substances and/or elements present.	Incorrect identification or incomplete identification.

If there is a discrepancy between the expected results and the experimental results, the supervisor must notify the Quality Assurance Manager. The Section Chief must begin an investigation and complete a *Corrective Action Request Form* (See ASCL-FORM-08). When complete, this form becomes part of the proficiency case file.

Each supervisor shall maintain a log of proficiency testing in each chemist's Employee History Binder.

3. 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 LIMS-plus program. Case reviews are documented on *Case Review Form* (ASCL-FORM-05).

If a reviewer discovers an error in the case record, the reviewer must document the error on the *Case Review Form* and inform the analyst. If the analyst and reviewer cannot reach consensus, then both the analyst and reviewer must meet with the supervisor for resolution.

If the error requires the analyst to correct administrative and/or examination records, the original record will remain in the electronic case file and the corrected record stored with a different name (i.e. corrected notes, corrected data, etc.).

4. Testimony Review

Refer to *ASCL Quality Manual* (ASCL-DOC-01) Section 5.9.6

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5.10. Reporting the Results

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 consists three columns of information: "Items", "Evidence Description" and "Test Results". When applicable, the body may also include statements about evidence sampling or disclaimers that aid the customer in understanding the report.

The general requirement for laboratory reports is that: The results of each test 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 supervisor to determine an approved method for reporting the information.

1. Guidelines

- A. All items (exhibits) documented in the chemist's case notes, including items not tested, should be included on the report.
- B. For items (exhibits) that *were not tested*³³ the report should include:
 - i. The item (exhibit) number,
 - ii. a description of the item,
 - iii. the phrase "not tested" either as part of the description or test results, and
 - iv. the exhibit's gross or net weight, as recorded in the case notes, with units and measurement of uncertainty.
 - v. Items excluded from testing in controlled substance manufacturing cases may be listed as a group at the end of the report and normally will not require a gross or net weight.
- C. For items (exhibits) that *were tested*³⁴ the report should include:
 - i. The item (exhibit) number.
 - ii. A description of the item.

³³ This includes items that may have been weighed or color tested to gain the information necessary to determine which items would be selected for testing.

³⁴ This includes tablets or capsules that have been pharmaceutically identified but were not analytically tested.

- iii. One or more values of initial exhibit amount³⁵, as recorded in the case notes, with units and measurement of uncertainty.
- If the substance was analyzed by rinsing the object, it must be reported as a “residue” or “no visible residue”. If the substance was weighed by the analyst but weighed less than 10.0 mg, it must be reported as “less than 10 mg”, or some similar terminology.
 - Weights should be reported in grams or kilograms and indicate the type of measurement they represent (i.e. net weight, gross weight, calculated net weight).
 - Counts should indicate the type of measurement they represent (i.e. count, calculated count).
 - Both weight and count must be reported for tablets or capsules containing a controlled substance³⁶.
 - The gross mass (carrier plus drug) should be listed for exhibits that tested positive for a controlled substance and a carrier was used to administer the substance (e.g. LSD and blotter paper).
- iv. The identities of elements, compounds or substances that were positively identified. Only elements, compounds or substances meeting the criteria of section 5.4.3. parts 1.C. & 1.D. may be reported.
- For compounds meeting the above criteria, a salt/base form or stereoisomer may be reported based on a positive or indicative IR spectrum.
 - If testing positively identifies a set of stereoisomers in an item but does not positively identify the specific isomer present, the results may be reported as *stereoisomer a/stereoisomer b*.³⁷
 - For tablets or capsules pharmaceutically identified to contain only non-controlled active ingredients and receiving no further analysis, the active ingredients, and if desired the dosage, will be reported preceded by the phrase “identified* as.” (see also requirement ‘e’ below).
 - For tablets or capsules pharmaceutically identified to contain controlled active ingredients and receiving further analysis, the positively identified³⁸ compounds will be reported. Additionally, if all pharmaceutically identified controlled ingredients have been positively identified, then all

³⁵ For evidence that involve manufacturing charges, the chemists will use their training and experience to determine if a sample should be weighed.

³⁶ Whether determined by pharmaceutical identification or analytical testing.

³⁷ e.g. ephedrine/pseudoephedrine.

³⁸ i.e. one positive category ‘A’ test for non-controlled substances and controlled substances.

the active ingredients, and if desired the dosage, may³⁹ be reported separately preceded by the phrase “identified* as” even if these ingredients include non-controlled substances not positively identified⁴⁰ during the analytical testing. (see also requirements ‘e’ below)

- e. All reports having results listed for tablets or capsules based on their pharmaceutical identification and preceded by the phrase “identified* as” will include the following disclaimer between the report’s last evidence item and the analyst’s signature: “*The identification results were obtained by comparing the item’s code imprint to imprint records and not by analytical testing. Any results confirmed by analytical testing are listed separately.”
- f. For sealed, intact commercially available pharmaceutical products (e.g. ampules, patches, etc.) identified based on their manufacturer’s label and receiving no further analysis, the active ingredients, and if desired the dosage, will be reported preceded by the phrase “identified* as.” (see also requirement ‘h’ below)
- g. For sealed, intact commercially available pharmaceutical products identified to contain controlled active ingredients and receiving further analysis, the positively identified⁴¹ compounds will be reported. Additionally, if all pharmaceutically identified controlled ingredients have been positively identified, then all the active ingredients, and if desired the dosage, may⁴² be reported separately preceded by the phrase “identified* as” even if these ingredients include non-controlled substances not positively identified⁴³ during the analytical testing. (see also requirements ‘h’ below)
- h. All reports having results listed for sealed, intact commercially available pharmaceutical products based on their manufacturer’s label and preceded by the phrase “identified* as” will include the following disclaimer between the report’s last evidence item and the analyst’s signature: “*The item(s) as received were sealed, commercially available

³⁹ If one or more of the active non-controlled ingredients in the preparation are not positively identified and the schedule of one or more of the controlled substances can vary depending on whether it exists in combination with non-controlled active ingredients, this “may” becomes a “must.”

⁴⁰ i.e. one positive category “A” test.

⁴¹ i.e. one positive category ‘A’ test for non-controlled substances and controlled substances.

⁴² If one or more of the active non-controlled ingredients in the preparation are not positively identified and the schedule of one or more of the controlled substances can vary depending on whether it exists in combination with non-controlled active ingredients, this “may” becomes a “must.”

⁴³ i.e. one positive category “A” test.

pharmaceutical items from a recognized manufacturer, in good condition, and without any apparent tampering. The identification results were obtained from the manufacturer's product labeling. Any results confirmed by analytical testing are listed separately."

- i. If no element, compound or substance was positively identified, the results may be reported as "no controlled substances detected."
- j. 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.
- k. If quantitative results will be reported, the compounds salt/base form should be reported if known or alternatively the compound identity may be anteceded by the phrase "as the *salt form* (or base)" where the chemist specifies the specific salt/ base form the quantitative value represents.
- l. Use TABLE 5.10-1 to determine the proper method to report results which may be significant in manufacturing cases.

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TABLE 5.10.-1 Reporting Results Significant to Manufacturing Cases

Result Reported	Necessary Tests
Phosphorus/Iodine	XRF
Inorganic salts (e.g. Ammonium nitrate, Sodium phosphate etc.)	IR solid ⁴
Ammonia ¹	IR ⁴ vapor, Nessler's
Lithium ²	IR ⁴ solid, Flame Test ♦
Lithium metal	IR ⁴ solid, Flame Test, Reactive w/ H ₂ O
Sodium ³	XRF, IR ⁴ solid
Sodium metal	XRF, Reactive with H ₂ O

¹ The following disclaimer must be added between the report's last evidence item and the analyst's signature: "The presence of ammonia cannot support the conclusion that anhydrous ammonia must have been present."
² The following disclaimer must be added between the report's last evidence item and the analyst's signature: "The presence of lithium cannot support the conclusion that elemental lithium must have been present."
³ The following disclaimer must be added between the report's last evidence item and the analyst's signature: "The presence of sodium cannot support the conclusion that elemental sodium must have been present."
⁴ If the IR results are positive, the substance is reported. If the results of the IR are indicative, the substance must be reported by its identity and anteceded by "indicated" (e.g. "ammonia indicated").

v. Quantitative results (if applicable)⁴⁴

a. Section 5.4.5.5. part 2.E. covers the calculation of quantitative results for reporting. Report the calculated percent purity truncated to three (3) significant figures with the following exceptions

- If the calculated percent purity exceeds 95%, report the purity as 95%.
- If the calculated percent purity is below 1%, report the purity as "less than 1%."

b. The Measurement of Uncertainty⁴⁵ for quantitative analysis is $\pm 5\%$ relative. Multiply the quantitative result by 0.05 to determine the absolute uncertainty to report⁴⁶.

e.g. The quantitative result for an item is 80.0% methamphetamine hydrochloride. Multiply 80.0% by 0.05, and round the result up using (2) significant figures to obtain the absolute uncertainty (i.e. 4.0). Report the result as: 80.0 (± 4.0) % methamphetamine hydrochloride.

⁴⁴ See also this section vi.b., second bullet.

⁴⁵ The MoU calculations and budget are located on the drug S:\SOP Clarifications\MoU documents.

⁴⁶ If the calculated percent purity is at the upper (i.e >95%) or lower end (e.g. <1%) of the reporting range, report the MoU as 4.8% for values above 95% and 0.050% for values below 1%.

- vi. Additional Reporting Requirements Related to Sampling, Sampling Plans and Sample Selection (if applicable)
- a. If any of the following situations apply, the chemist will consult the appropriate supervisor to determine the appropriate language for reporting the information.
 - b. When sample selection is used, the report must state what was received, what was tested, and must be clear that the result/conclusion pertains to that which was tested.
 - For multi-unit populations composed of solid dosage forms (e.g. tablets, capsules, LSD on blotter paper squares etc.) the report for the item must also include a statement indicating what portion of the whole constituted the portion that was tested.
 - For all other multi-unit populations, when only a portion of the population is tested the report must include:
 - A description of the entire population including a gross 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 of what was not tested, the total gross weight of the items not tested, and the phrase “not tested” either as part of the description or in the test results column.
 - c. 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.

2. Editorial Correctness

After the report is generated, the chemist will proof the following areas of the report:

A. Header

Information in the header (e.g. Investigating Officer/Agency/Address, Suspects(s), Laboratory Case Number, etc.) should be compared to the case's *Evidence Submission Form* for completeness, misspellings and incorrect information. If necessary, the chemist will correct the information in LIMS-plus so the header matches the information on the *Evidence Submission Form*.

B. Analytical Results

The chemist should compare the report body (items, descriptions, test results etc.) to the information in their case notes for discrepancies. If necessary, the chemist will correct the results to match the information in their case notes.

C. Signing Reports

The chemist will electronically sign their report after the proofing process is complete. A report will be issued to the investigating agency.

3. Supplemental and Amended Reports

If the analysis associated with a supplemental request is completed by a chemist other than the one assigned to the original request then consult the appropriate supervisor for instructions on reporting the results.

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