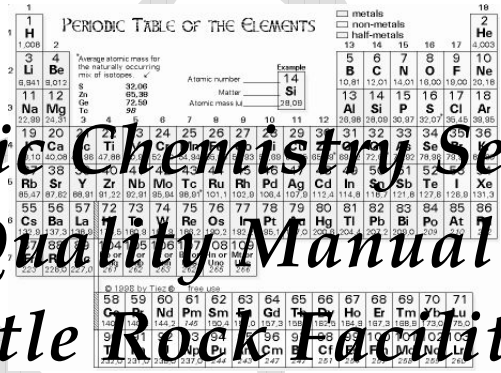
Arkansas State Crime Laboratory



Forensic Chemistry Section Quality Manual Little Rock Facility

Executive Director:
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1. Introduction

This manual contains the policies and procedures outlining the general operation of the Forensic Chemistry Section of the Arkansas State Crime Laboratory. All Forensic Chemists are responsible for reviewing, knowing, understanding and following these guidelines. This policy and procedure manual should be considered a living document requiring constant review by the Forensic Chemistry staff. Policies and procedures may become obsolete and/or outdated and may require revision, replacement or elimination. This manual will be updated as changes or additions occur.

The Forensic Chemistry Section analyzes evidence to provide law enforcement officers and the judicial system with 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. (Evidence is defined as items submitted by law enforcement agencies to serve the purpose stated above.). 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.

Forensic Chemists may also be called upon to assist law enforcement agencies in the dismantling of suspected illicit laboratories, to collect representative samples of evidence, and to submit the samples to ASCL Evidence Receiving Section on behalf of the law enforcement agency.

2. Personnel Qualifications and Training

2.1 Educational Qualifications

Forensic Chemists must possess the formal education equivalent of a bachelor's degree in chemistry and three years experience in a chemical laboratory. 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.

2.2 Special Training Requirements

Details of the training requirements may be found in Forensic Chemist Training Manual but are described briefly as follows: Each Forensic Chemist trainee must complete an initial in-house training period of twelve weeks (minimum) on general drug analysis. After successfully completing the initial training, the trainee will be required to successfully complete competency tests (including a written test), internal proficiency tests, and moot court, prior to performing independent casework. The mock trial requirement may be waived if the trainee has documented participation in previous mock trial(s) or has previous experience testifying as an expert witness. If this requirement is waived, the section chief must provide appropriate documentation in the *Employee History Binder*.

Following training in basic drug analysis, all personnel who will have the responsibility of processing suspected off-site clandestine laboratories must complete the following training steps:

1) Successfully complete an in-house training module on advanced clandestine laboratory analysis as listed in the Forensic Chemist Training Manual.

2) Pass a full physical and complete a one (1) week certification course in clandestine laboratory investigation provided by the DEA.

3) Upon completion of the DEA certification course, trainees will participate in the processing of suspected clandestine laboratories while supervised by a qualified Forensic Chemist. Approval to independently process suspected clandestine laboratories will be given by the Chief Illicit Labs Chemist. Approval will be based on evaluation of verbal and/or written reports from qualified Forensic Chemists who have supervised the trainee in the field.

3. Facilities

Security of Forensic Chemistry Section

The toxicologists, forensic chemists, trace and physical evidence analysts have 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 Forensic Chemistry Section Chief also has access to these storage areas.

3.1 Security

1. Security of Primary Standards

A. Access

The primary (controlled substances) standards are kept in a locked filing cabinet in the office of the Chief Forensic Chemist. The keys to the locked filing cabinet are assigned to Chief Forensic Chemist, Chief Forensic Toxicologist, and Chief Illicit Labs Chemist. The Forensic Chemistry Chief may designate other personnel as needed.

The Chief Forensic Chemist or designee is responsible for ordering new primary standards and for logging them into the Controlled Substance Logbook upon their receipt. The logbook should show the initials, date received, the amount received, the lot number(s), and the balance. Table 1 lists the checkout procedure for primary standards. [**NOTE:** Before the primary standards are used in casework, they must be verified (see Section 6.5-3 Part 3A (i) — Purchased Standards.)]

TABLE 1 *Checkout Procedure for Primary Standards*	
Step 1	Record initials, date, standard number, and standard name on the Chronological Primary Standard Log Sheet.
Step 2	Retrieve the primary standard from the locked drug cabinet.
Step 3	Obtain an accurate measurement of the amount used.
Step 4	Return the primary standard to the locked drug cabinet.
Step 5	Record the amount on the Chronological Primary Standard Log Sheet.
Step 6	Obtain initials of the employee responsible for locking the cabinet on Chronological Primary Standard Log Sheet to verify return of the

	primary standard.
Step 7	In the Controlled Substance Logbook, document your initials, the date, and the amount used.
Step 8	Subtract the amount used from the old balance and record the new balance in the Controlled Substance Logbook.

B. Annual Inventory

State and Federal law require an annual inventory of all primary standards. The Chief Forensic Chemist is responsible for this inventory. Table 2 lists the procedure for performing an inventory of the primary standards.

TABLE 2 ***Annual Inventory of Primary Standards***	
Step 1	Obtain an accurate measurement of the primary standard.
Step 2	Enter this measurement, the date, your initials and the notation "inventory" in the Controlled Substance Logbook.
Step 3	Repeat Steps 1 and 2 for all primary standards.
Step 4	Prepare an inventory summary that includes the standard name, standard number, date(s) the inventory was performed, identification of chemist(s) performing inventory, Logbook balance, inventory weight, and any discrepancies.
Step 5	Provide a copy of the summary to the Quality Assurance Manager and the Forensic Chemistry Section Chief.

C. Disposal

A primary standard may be disposed of as deemed appropriate by the Forensic Chemistry Section Chief. Table 3 lists the procedure for disposing of a primary standard.

TABLE 3 ***Destruction of Primary Standards***	
Step 1	Obtain an accurate measurement of the primary standard.
Step 2	Enter this measurement, the date, your initials and the notation "inventory" in the Controlled Substance Logbook.
Step 3	Seal, initial, and date the primary standard.
Step 4	Fill out the appropriate paperwork for the Arkansas Department of Health.
Step 5	Transport the primary standard and the paperwork to the Arkansas Department of Health for destruction.

Step 6	Document the amount of standard disposed, the date, your initials, and the notation "disposed" in the Controlled Substance Logbook.
Step 7	The paperwork is filed in the folder marked "Destruction-Controlled Substance Standards/Vegetable Material Waste" located in the office of the Chief Forensic Chemist.

2. Security of Secondary Standards

Secondary standards may be obtained to aid the chemist in the analysis of an evidence exhibit. Chemists may retain a portion of an evidence item for addition to the secondary standard inventory to be used as a standard in future case work, for training purposes or for potential use as a proficiency sample.

The secondary standards to be used in casework will be kept in a locked filing cabinet in the office of the Chief Forensic Chemist and will be logged into the Secondary Standard Logbook upon their receipt. The logbook should show the initials, date received, the amount received, standard origin, and the balance. An annual inventory must also be completed.

The secondary standards intended for training purposes or for potential use as a proficiency sample must be kept in a secure storage cabinet selected by the Chief Forensic Chemist and will be logged into the Secondary Proficiency Standards Logbook upon their receipt. The logbook should show the initials, date received, the amount received, standard origin, and the balance. An annual inventory must also be completed.

Table 4 lists the procedure for obtaining an annual inventory of secondary standards.

TABLE 4 ***Annual Inventory of Secondary Standards***	
Step 1	Obtain an accurate measurement of the secondary standard.
Step 2	Repeat Steps 1 and 2 for all secondary standards.
Step 3	Prepare an inventory summary that includes the standard name, date(s) the inventory was performed, inventory weight, and any discrepancies.
Step 4	Provide a copy of the summary to the Quality Assurance Manager and the Forensic Chemistry Section Chief.

3. Security of Controlled Substance Waste

After analysis, the chemist should dispose of any remaining sample in the appropriate waste container (e.g. vegetable material into cans marked waste,

powders into chemical waste bottles). The vegetable material waste must be collected on a regular basis, heat-sealed in a plastic bag, marked with gross weight and initials, and then locked in a secure storage area until disposed of. Table 5 lists the procedure for disposing of vegetable material waste.

TABLE 5 *Destruction of Vegetable Material Waste*	
Step 1	Fill out the appropriate paperwork for the Arkansas Department of Health.
Step 2	Transport the vegetable waste and the paperwork to the Arkansas Department of Health for destruction.
Step 3	The paperwork is filed in the folder marked "Destruction-Controlled Substance Standards/Vegetable Material Waste" located in the office of the Chief Forensic Chemist.

Chemical waste bottles are located in several of the hoods in the Forensic Chemistry Section. The waste bottles are regularly transported to the basement and their contents transferred to waste barrels for storage and disposal.

4. Evidence Control and Case Management

Purpose: This document establishes general standards for case management and evidence control by the Forensic Chemistry Section, in addition to policies documented in the ASCL Quality Manual.

Scope: This section states the evidence examination and case priority policies along with the chain of custody of evidence, evidence storage, and evidence seals and markings. Deviations from this policy should be approved by the section supervisor or designee.

Evidence Examination Policy

The ASCL Quality Manual states, "The laboratory may discontinue further forensic examinations when a conclusion identifies, includes or eliminates the subject(s) or substantiates the maximum charge to be filed" (see ASCL Quality Manual, Section 4.2). The Forensic Chemistry Section's application of this policy is dependent on the type of submission and/or the charges filed and will be addressed in Section 6.3 (Evidence Sampling Policy) of this manual.

Case Priority Policy

Chemists in the Forensic Chemistry Section are encouraged to process the oldest cases first. Exceptions may occur when subpoenas are issued with the date of the trial/hearing being sooner than the case would normally be processed, or at the officer's/prosecutor's request.

Chain of Custody

It is the responsibility of the analyst to maintain proper control of all evidence in their possession. Analysts in the Forensic Chemistry Section will use the south elevator or stairway (when available) to transport evidence to and from the Evidence Receiving Section. Each chemist will personally receive evidence from an Evidence Receiving Technician 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 the Evidence Receiving Section. Evidence may be transferred between analysts, as deemed necessary. The section chief may set limits on the number of cases checked out or retained in an analyst's storage area(s).

Evidence Storage

Each analyst in the Forensic Chemistry Section is assigned lockable storage areas to be used for short term storage of evidence. Keys to the storage area(s) are issued to the

analyst. A duplicate key is stored in a locked key cabinet located near the door of the Forensic Chemistry Section Chief's office. The keys to the locked key cabinet are under the control of the Forensic Chemistry Section Chief and the Executive Director.

If it is necessary to reassign evidence, 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.

During the sampling of evidence, it may be necessary to have unsealed evidence in the Forensic Chemistry Section (a limited access area). This time should be kept to a minimum and resealing 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 Forensic Chemistry Section for any extended period of time.

Evidence may be stored for short periods of time in the hood when drying is necessary or when there are safety concerns. 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).

Evidence Seals and Markings

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. Items of evidence or their proximal containers should be marked with the unique ASCL case number (YYYY-00000), item number and the analyst's initials. When heat-sealing, the analyst's initials will be made across each heat-seal. On the outside packaging, the analyst should place their initials across their seal.

Miscellaneous

All exhibits in a case should be compared to the submission sheet. Significant discrepancies should be discussed with the investigating agency.

Unattended, automated analysis of evidence is permitted as long as the samples and instrumentation involved remain in a limited access area.

Sampled evidence not consumed in analysis should be disposed of in containers labeled "Waste."

5. Validation

The Forensic Chemistry Section uses procedures that are well established and generally accepted in the field of chemical analysis. In Section 6.6 Testing Techniques, references are provided to document that the procedures used are based on widely accepted scientific principles.

The established calibration curves used for quantitation by gas chromatography are subjected to on-going validation by the use of check samples. The check samples are controls that are performed daily before the calibration curve can be used for casework. The printouts for the check samples are kept in a binder near the instrument.

Before a new procedure can be used for case work, the procedure must be validated. TABLE 5 shows the steps necessary to validate a new procedure.

TABLE 5 ***Validation Requirements***	
Step 1	Document the theoretical basis for the procedure.
Step 2	Assess the specificity of the procedure.
Step 3	Assess the limitations of the procedure.
Step 4	Predict possible sources of error.
Step 5	Test the procedure using known samples.
Step 6	If practical, compare split samples between an established method and the new method.
Step 7	Test known samples for effects of such factors as matrix, sample age, degradation…etc.
Step 8	If the procedure provides quantitative data, include estimations of accuracy and precision at concentrations representative of casework.
Step 9	Conduct a validation study using split samples, blind trials, and/or concordance testing
Step 10	Submit Steps 1 through 9 to the appropriate Section Chief for approval.

All documentation supporting validation must be kept on record. After a new procedure has been validated, it should be incorporated into the Forensic Chemistry Quality Manual.

6. Analytical Procedures (SOP)

6.1 Notation Procedures

Purpose: These procedures establish general standards for case notes and supporting examination documentation by the Forensic Chemistry Section for the Arkansas State Crime Laboratory (ASCL) in addition to the policies documented in the ASCL Quality Manual, Section 9.1.

Scope: This section defines contents that are required to be documented in the analyst's case notes. Deviations from these guidelines must have the approval from the Forensic Chemistry Section Chief or designee.

1. **Abbreviations**
   Each forensic chemist will submit a legend of his/her commonly used personal abbreviations to the Forensic Chemistry Section Chief or designee for approval. After being approved, each chemist's abbreviation legend will be maintained in the *Employee Abbreviations Binder* which is accessible to reviewing chemists.

2. **Requirements for Handwritten Notes and Observations**
   The Arkansas State Crime Laboratory is currently using the JusticeTrax LIMS-plus software program. All case documentation, including Handwritten Notes, will be stored electronically.

   A. Handwritten notes and observations must be in ink. However, pencil may be appropriate for diagrams or making tracings.

   B. Nothing in the handwritten information will be obliterated or erased. Any corrections will be made by an initialed (handwritten), single strikeout (so that what is stricken can still be read). Correction fluid or correction tape may not be used.

   C. Additional notations, including interlineations, made in case notes must be initialed (handwritten) by the person making the additions.

   D. The first page of the notes must include the total number of notes pages by either using the method of "page 1 of x" or by including a statement that the notes contain x number of pages.

E. Each page of the case notes must include…
   i. The unique ASCL case number (YYYY-00000),
   ii. handwritten examiner's initials[1],
   iii. 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.),
   iv. and page number.

F. Every tested 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 Forensic Chemist could identify the evidence based only on their notes. If repackaging of the item is necessary, the analyst will indicate this in the notes. Evidence that is not tested in accordance to Section 6.31Aiii and is properly described on the evidence list or submission sheet does not require re-description in the case notes. However a list of the items not tested must be included in the notes, at any point and in any manner the chemist chooses.

G. Most items which are tested are required to have a measurement of their amount (i.e. weight, volume and count) taken before and after sampling. These initial and reserve measurements must be documented in the case notes (see Section 6.4, Recording and Reporting Measurements, for specific procedures).
   i. For items containing tablet(s)/capsule(s) pharmaceutically identified to contain a non-controlled substance(s) and are not sampled for further testing, the Forensic Chemist must document the count (total number of tablet(s)/capsule(s)) in the case notes.
   ii. For items containing tablet(s)/capsule(s) pharmaceutically identified to contain a controlled (or penalty) substance(s) and are sampled for further testing, the Forensic Chemist must document…
      a. the count (total number of tablet(s)/capsule(s)) in the case notes,
      b. the initial and reserve weights[2] of the tablet(s)/capsule(s),
      c. and the number of whole and/or partial tablet(s)/capsule(s) sampled.
   iii. For items containing tablets(s)/capsule(s) that are not of pharmaceutical origin and are sampled for further testing, the Forensic Chemist must document…
      a. the count (total number of tablet(s)/capsule(s)) in the case notes,

[1] 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.
[2] Sealed containers of tablet(s)/capsule(s) (e.g. sealed blister packs) are excluded from the weight measurement. The tablet(s)/capsule(s) will be counted only.

b. the initial and reserve weights of the tablet(s)/capsule(s),
c. and the number of whole and/or partial tablet(s)/capsule(s) sampled.

iv. Items that cannot be measured are to be listed as residue in the case notes and on the report if a visible substance is present.

v. Forensic chemists may exclude wet solids from the measurement requirement with approval of the supervisor.

vi. Measurements are taken the day the evidence is sampled unless otherwise notes.

H. If the chemist has to homogenize the item before a sample is taken, they must document…

i. an approximation of the amount of the item prepared (e.g. ~½, ¾ etc.),

ii. and the method of preparation (e.g. grinding.)

I. During testing, the Forensic Chemist must document in their case notes…

i. the tests performed,

ii. the date on which the tests were performed unless the instrumental documentation contains an instrument generated date (this applies to FTIR, GC, GCMS and XRF),

iii. and the results of the tests.

J. If an item requires a solvent extraction, the Forensic Chemist must document in their case notes…

i. a description of the extraction procedure used,

ii. and the date the extraction was performed.

K. If the chemist has to take a core sample, it must be documented in the case notes.

L. Any other notations required for an individual testing technique are stated in the appropriate subsection of Section 6.6.

3. Requirements for Supporting Examination Documentation

The Arkansas State Crime Laboratory is currently using the JusticeTrax LIMS-plus software program. All supporting examination documentation will be stored electronically. Each page of the supporting examination documentation must include:

i. The unique ASCL case number (YYYY-00000),

ii. the exhibit number,

iii. the date(s) the examination(s)[3] was performed.

[3] For documentation that is not an examination (i.e. reference material) the date documented will be the date that the material was generated.

6.2 Testing Requirements

Purpose: This document describes the testing requirements for examination of evidence in the Forensic Chemistry Section.

Reference(s):

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986.

U. S. Department of Justice, Drug Enforcement Administration (DEA). *Basic Training Program for Forensic Drug Chemists*.

SWGDRUG: *Recommendations for Education and Training, Quality, Assurance, Methods of Analysis*; distributed 11/01

Scope: This document provides an overview of policies and procedures used for the analysis of evidence exhibits. Analysis protocols must be flexible and general in nature, due to the extreme variety of evidence submissions.

1. **Qualitative Analysis**

   A. General Specificity for the Results of Common Testing Techniques

TABLE 6 ***Categories for Common Testing Techniques***		
Category A	Category B	Category C
Infrared Spectrometry (IR) Gas Chromatography/Mass Spectrometry (GC/MS)	Gas Chromatography (GC) Thin-Layer Chromatography (TLC) Pharmaceutical Identifiers	Color Tests

   B. Quality of Test Results

   The results of a category "A" or "B" test may be positive, indicative or negative for a compound based on the quality of those results. Criteria for considering a test to be positive for a compound will be addressed in each test's technique description (see Section 6.6 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 Forensic Chemist are usually indicated by the

type of sample. Category "C" tests are normally used to help plan future testing of the sample and for determining the possibility of combining item contents within exhibits composed of multiple items.

C. Minimum Testing of Exhibits

i. Minimum Tests per Exhibit

For exhibits requiring sampling and testing (as specified in Section 6.3 Evidence Sampling Policy), the Forensic Chemist must perform, at a minimum, two tests per exhibit in order to report *Test Results* for that exhibit on the *Report of Laboratory Analysis* generated at the conclusion of testing[4]. 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 is the preferred category "A" test.

ii. Minimum Tests per Exhibit: Exceptions

a. Items that test positive for tetrahydocannabinol (THC) or marihuana (as defined below in Section 6.2 Parts 1Diia & b) are normally excluded from further testing.[5,6]

b. Pharmaceutical tablet(s)/capsule(s) which have been pharmaceutically identified to contain only non-controlled substances are normally excluded from further testing unless tampering is apparent or suspected.

c. Samples taken for elemental analysis by X-ray Fluorescence 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 Section 6.2 Parts 1D (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

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

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

[6] Items that also contain powder material must be subjected to a minimum of two additional tests as stated in Section 6.2.1.C.i.

testing. Proceed to Section 6.2 Part 1D (iv) to determine if the minimum requirements for identifying a non-controlled compound have been met.

c. Items suspected to contain marihuana and/or tetrahydrocannabinol (THC) that do not meet the identification requirements of Section 6.2 Parts 1Diia & b must be subjected to GCMS testing.

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

a. Positive tetrahydocannabinol (THC) identification requires a positive gas chromatography mass spectrometry (GCMS) result and one positive result from the following:
   - thin-layer chromatography (for THC),
   - modified Duquenois-Levine (for cannabis)
   - qualitative gas chromatography with retention time(GC)

b. Positive marihuana identification requires positive results from a microscopic examination (for cystolithic hairs) and positive results for two of the following tests:
   - thin-layer chromatography (for THC),
   - modified Duquenois-Levine (for cannabis),
   - qualitative gas chromatography with retention time(GC)
   - gas chromatography mass spectrometry (GCMS)

iii. Controlled or Penalty Compounds: Additions

a. For positive identification of pseudoephedrine/ephedrine, infrared spectrometry testing must be conducted[7] on the sample in order to distinguish between the two compounds[8].

b. Exhibits, excluding residues, in which cocaine has been detected, must be analyzed by infrared spectrometry so that, if possible, the cocaine form (i.e. salt or base) may be determined.

iv. Elements & Non-controlled Compounds

[7] Results of the IR testing must be either positive or indicative for pseudoephedrine or ephedrine.

[8] This requirement is waived for positive identification of pseudoephedrine/ephedrine in tablet(s)/ capsule(s) 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 spectroscopy.

a. The analyst may reach a positive conclusion on the presence and identity of a non-controlled substance based on one positive category "A" test.
b. 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.
c. 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,
   - and an X-Ray Fluorescence spectra of the sample showing the sample to be negative for the presence of elements known to produce colors similar to lithium with a flame test[9].
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

TABLE 7 *Arkansas Rebuttable Presumption Limits*[1]	
Drug	**Amount**
Heroin	100 milligrams
Morphine	300 milligrams
Cocaine	1 gram
Codeine	300 milligrams
Pethidine (Meperidine)	300 milligrams
Hydromorphine Hydrochloride	16 milligrams
Methadone	100 milligrams
Lysergic Acid Diethylamide (LSD)	100 micrograms
Stimulant Drug	200 milligrams
Ephedrine	5 grams
Pseudoephedrine or phenylpropanolamine	9 grams
[1] Taken from Arkansas Code 5-64-401 and 5-64-1101.	

A. Exhibits For Which a Quantitation are Required

[9] Strontium produces a red flame.

Exhibits, in which one of the drugs listed in TABLE 7 is detected according to the requirements in Section 6.2 Part 1, should be quantitated when it may determine the schedule of the drug or when there are special circumstances. (Exception: If there is not enough sample to perform a quantitation on the substance, it may be omitted for purity testing. (i.e. codeine quantitation procedure requires 5mL of sample)) The chemist will quantitate the sample if it has been confirmed to contain at least of five (5) grams of ephedrine or nine (9) grams of pseudoephedrine or phenylpropanolamine.

B. Exhibits For Which a Quantitation are Required: Exceptions

i. In cases containing tablets that are intact and have been pharmaceutically identified to contain a known amount of one or more drugs listed in TABLE 7, these tablets are exempt from quantitation[10].

[10] Pharmaceuticals in liquid form containing one or more of the drugs from TABLE 7 may also be exempt from the quantitation requirements if they are in a sealed condition. The supervisor will make a decision on a case by case basis.

6.3 Evidence Sampling Policy

Purpose: This section establishes general guidelines for evidence sampling by the ASCL Forensic Chemistry Section.

Scope: This policy provides guidelines for the selection of evidence items and proper evidence sampling.

1. Selection of Evidence Items

A. General

i. The number and type of exhibits to be sampled and tested will be determined by the submission and type(s) of offense.

ii. For controlled substance offenses[11] involving possession or delivery, all exhibits that are suspected of containing a controlled substance, with the possible exclusion of paraphernalia (see Section 6.3.1.D Drug Paraphernalia policy), must be sampled for testing.

iii. For controlled substance offenses[12] involving manufacture, the Forensic Chemist may choose to use the following scheme to determine which items will be sampled and tested regardless of the number of items submitted as evidence.

a. Based on their training and experience, the Forensic Chemist will select five (5) items, at a minimum, for sampling. The items which appear to present the highest probability of supporting a charge of manufacturing should be selected

b. Negative or inconclusive results for tests run on the selected items will necessitate the sampling and testing of additional items. In this case the Forensic Chemist will continue until either the highest charge is substantiated or items for testing are exhausted.

c. In addition to items selected to substantiate the charge of manufacturing, all exhibits containing powders[13] in appreciable (weighable) amounts will be sampled and tested by the Forensic Chemist.

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[11] i.e. Possession of Drug Paraphernalia, Possession of an Instrument of Crime, Possession of a Controlled Substance, Possession of a Controlled Substance with Intent to Deliver, Delivery of a Controlled Substance.

[12] i.e. Possession of Drug Paraphernalia with Intent to Manufacture a Controlled Substance, Conspiracy to Manufacture a Controlled Substance, Manufacture of a Controlled Substance.

[13] If in the Forensic Chemist's opinion, any powder item(s) subject to this provision would only be subjected to elemental analysis and/or only likely to produce test results identical to an item(s) already selected for testing then the item(s) meeting these criteria may be excluded from testing.

d. All remaining items that have no bearing on the highest charge to be substantiated may be excluded from sampling by the Forensic Chemist.

B. Multiple Items in an Exhibit

If multiple items are present in a single exhibit, the items may be combined for testing if the following criteria are met:

i. All items have the same physical appearance (e.g. E1 consists of ten ziploc bags each containing powder with the same color and consistency).

ii. Examination of samples from each item by a common test from Category "A", "B", or "C" (see Section 6.2 — Testing Requirements) produces identical results (e.g. samples from each of the ten ziploc bags are treated with Marquis Reagent and produce the same color).

iii. The items' contents are not vegetable material.

C. Tablets/Capsules

i. Non-controlled tablets/capsules

If a tablet/capsule is pharmaceutically identified (using the color, shape, imprint and/or scoring of the tablet/capsule) as a non-controlled substance, then a sample does not need to be taken for analysis.

ii. Controlled and penalty tablets/capsules

If a tablet/capsule is pharmaceutically identified (using the tablet/capsule imprint and any of the following: color, shape, scoring etc.) as containing a controlled substance or penalty drug then a sample must be taken for analysis (see Section 6.3 Part 2C).

iii. Other tablets/capsules

If a pharmaceutical identification on a tablet/capsule cannot be made or it is suspected that tampering may have occurred, then a sample must be taken for analysis (see Section 6.3 Part 2C).

iv. Identification, sampling and testing of tablets/capsules may be omitted in manufacturing cases (see this section, 1A) unless their presence contributes to proving the manufacturing charge.

D. Drug Paraphernalia

Drug paraphernalia shall be tested if it is the only drug evidence present in the case and/or is needed to satisfy a charge. Drug paraphernalia (including syringes) may be excluded from testing in the following circumstances:

i. In the presence of other drug items in the same case such as powders, vegetable material, or other actual drug evidence with positive laboratory test results to satisfy a possession, sale, or manufacture charge.

ii. When the paraphernalia presents a safety hazard to the analyst. This includes syringes as well as broken glass or other objects that might cause injury from accidental needle sticks or cuts.

This policy is intended to help expedite cases in a timely manner and provide the necessary laboratory results for prosecution of drug cases.

E. Other

Items listed as probable cause for a search should be analyzed.

Cross contamination of items may preclude the examination of the contaminated items(s).

2. Proper Sampling of Evidence

When sampling, the analyst must make every effort to preserve a portion of the evidence item.

A. Solids, Powders & Vegetation

A representative sample suitable for analysis should be taken. Each unique appearing part of the material may be collected and analyzed together or separately[14]. For a (non-quantitative) compressed sample (i.e. brick), a core sample must be taken.

*Items to be **quantitated*** must be homogenized before sampling.

i. For a non-compressed sample, a minimum of 50% of the sample should be ground and mixed. When placing the reserve sample back into the case, the unprocessed portion of the exhibit must be separated from the processed portion.

ii. For a compressed sample (i.e. brick), a core sample is acceptable for a representative sample. This representative sample must be ground and mixed. If a core sample is not taken, then a minimum of 50% of the sample should be ground and mixed. When placing the reserve sample back into the case, the unprocessed portion of the exhibit must be separated from the processed portion.

[14] For items containing both vegetable and powder material in the same sample, if possible the chemist will separate the two distinctive materials and test them separately.

B. Liquids

Liquid samples should be sampled directly into a screw top vial or covered test tube in order to avoid evaporation of the sample.

Single-layer liquids must be agitated before sampling. Multi-layer liquids require sampling of each layer. Liquids containing solid precipitates require sampling of the liquid and solid.

The method of sampling for quantitation will be determined based on the specific circumstances of the evidence.

C. Tablets/Capsules

If more than one tablet/capsule is present, each tablet/capsule must be visually observed to make sure that each tablet/capsule contains the same marking/imprints[15].

At a minimum, take the square root of the number of tablet(s)[16]/capsule(s) in the exhibit, randomly select this many tablet(s)/capsule(s) from the exhibit. Of the randomly selected tablet(s)/capsule(s),

i. If the tablet(s)/capsule(s) have been pharmaceutically identified, then retain half of each tablet, partial tablet, or approximately half of the capsule's contents for testing.

ii. If the tablet(s)/capsule(s) have not been pharmaceutically identified, then the forensic chemist will test each tablet, partial tablet, or according to the following:.

   a) For tablets that screen the same, retain half of each tablet, partial tablet, or approximately half of the capsule's contents for testing.

   b) For tablets that do not screen the same, all tablets in the item must be screened and grouped together as sub-exhibits according to their screening results. Each sub-exhibit will be sampled (taking the square root of the number of tablet(s)/capsule(s) in the sub-exhibit and retaining half of each tablet, partial tablet, or approximately half of the capsule's contents for testing) and tested separately.

[15] Exception: In an exhibit item that contains whole and partial tablet(s), if the partial tablet(s) contain similar markings as the whole tablets, they can be combined with the whole tablets for testing.

[16] Partial tablets must be counted in the total number of tablets (i.e. 3 whole tablets with 6 partial tablets; take the square root of 9, sampling whole and partial tablets.)

The method of sampling for quantitation will be determined based on the specific circumstances of the evidence.

D. Paraphernalia
Items of paraphernalia may be sampled by rinsing with a suitable solvent.

E. Suspected LSD
Representative samples should be taken from each suspected LSD exhibit. Suspected LSD samples should be sealed, marked and returned with the evidence when practical.

3. 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.

4. Reusable Glassware

If reusable glassware is used, the analyst will wash the selected glassware with an appropriate solvent. The analyst will then take a small amount of solvent and rinse the glassware. This will then be injected on the GCMS to ensure that the glassware does not contain any contaminants. This will be included in the appropriate electronic case file. If contaminant(s) are found and the analyst has used the glassware in casework, the sample must be re-tested.

6.4 Recording and Reporting Measurements

Purpose: This method describes the procedures used when counting or measuring (mass or volume) the amount of an exhibit received in the Forensic Chemistry Section.

Scope: This document describes guidelines on measuring items, in addition to calculating and reporting measurements in the Forensic Chemistry Section.

TABLE 8 ***REQUIRED EXHIBIT MEASUREMENT(S)***

Common Exhibit Classes	Amount(s) Measured	Measurement Reported
Solids[3] (powders, vegetation)	Weight	Weight
Liquids	Volume[1]	Volume
Tablet(s)/capsule(s)	Count Only in the following situation(s): ◎ the tablet(s)/capsule(s) (whole) have been pharmaceutically identified to contain a non-controlled substance.	Count
	Count & Weight in the following situation(s): ◎ the tablet(s)/capsule(s) have been pharmaceutically identified to contain a controlled or penalty substance[2], ◎ the tablet(s)/capsule(s) are not of a pharmaceutical origin, ◎ there is a suspicion that the tablet(s)/capsule(s) have been tampered with, or ◎ the tablet(s)/capsule(s) cannot be pharmaceutically identified.	Count and/or Weight
	Weight Only in the following situation(s): ◎ Exhibits containing partial, broken, or crushed tablet(s)/capsule(s).	Weight
Items suspected to be delivery vehicles for LSD (e.g. blotter paper, sugar cubes, candy, etc.).	Weight & Count (if appropriate)	Weight
Pharmaceutical patches	Count	Count
Solids suspected of being reaction sludge, pill "dough" or elemental iodine or phosphorus.	Normally excluded from any measuring requirements.	N/A

[1] For liquids in sealed pharmaceutically labeled containers, the label marking may be used for the initial volume measurement. [2] Tablet(s)/capsule(s) in sealed containers (e.g. sealed blister packs) are excluded from the weight measurement. The tablet(s)/capsule(s) will be counted only. [3] Wet solids may be excluded from the measurement requirement with the approval of the supervisor.

1. Weight (Mass) Testing & Reporting

A. Requirements for Weight Measurement

Select a suitable verified balance (see Section 7.2 Balances) for the item(s) to be weighed. Both an initial and reserve weight, in grams or kilograms, should be recorded in the case notes exactly as displayed on the balance. If the Forensic Chemist must use a balance other than their personal issue (e.g. another chemist's balance or the bulk scale), 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.

The analyst may acquire a net weight, a tared net weight or a calculated net weight for the item based on TABLE 9.

TABLE 9 ***Notation and Reporting of Acceptable Weight Types***

Weight acquired	**Notes required**	**Report**
Net weight	Initial and reserve net weight.	Initial net weight
Tared net weight[1, 2]	Packaging weight for item to be tared, tared initial net weight(s), tared reserve net weight(s).	Tared initial net weight
Calculated net weight[1]	Packaging weight for any item(s), all initial gross weights, reserve weight (gross and/or calculated net), all calculations.	Calculated initial net weight

[1] Packaging used for tares or for calculating net weights should follow the guidelines in 6.4 Part 1B (ii) Calculated Net Weights.

[2] If the tared packaging weight for an item is used for other items, it must be documented in the case notes which items used this tared packaging weight.

If the substance is less than an amount that can be weighed on the analytical balance, it will be listed as a residue in the notes and on the report. if a visible substance is present.

B. Calculation(s)

i. Total Weights

a. For a single exhibit containing multiple items with identical test results, the individual weights as recorded may be summed.

b. For consecutive multiple exhibits containing items with identical test results, the individual weights as recorded may be summed.

c. If multiple balances are used, the sum is truncated to reflect the accuracy of the least accurate balance.

ii. Calculated Net Weights

a. Calculated net weights may be used for items packaged in the same manner. In such cases, the weights of representative packaging samples are determined and used to make these calculations.

b. Calculated net weights should always be conservative to the extent that the analyst could testify that the calculated net weight is less than the actual net weight.

c. Rounding should not be performed.

2. Volume Testing & Reporting

A. Requirements for Volume Measurement

For liquids packaged in containers that are graduated, the volume may be measured and recorded based on the container's scale. Otherwise, the analyst must use a graduated container[17] to measure the volume of the liquid. All volumes will be reported in milliliters or liters. The device used to measure the volume should be documented in the case notes (i.e. 10mL graduated cylinder).

B. Calculation(s)

If multiple devices are used, all volumes are summed as recorded from the measuring device used, and the digits truncated to reflect the accuracy of the least accurate measuring device.

3. Counting & Reporting

A. Requirements for Count Measurement

The total number of tablet(s)/capsule(s) in an exhibit must be counted or calculated (see calculations below) and reported as that number of tablet(s)/capsule(s).

B. Calculation(s)

For exhibits containing more than one hundred pharmaceutical tablet(s)/capsule(s), the analyst may calculate the total number of tablet(s)/capsule(s) present in the exhibit using a weight proportion set up as follows:

[17] e.g. graduated cylinder, disposable syringe. In manufacturing cases, approximate volumes may be obtained by comparison of the liquid level in the evidence container to known volumes marked in a container of similar size and shape.

EQUATION 1 ***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.*

$n_{Count} \geq 100$, *The number of tablets/capsules counted.*

m_{Count} = *The mass of* n_{Count}.

Example: The Forensic 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.

The chemist counts out 100 tablets (n_{Count}). The 100 tablets are measured to have a weight of 10.2121 grams (m_{Count}).The chemist next measures the weight of the entire number of tablets to be 241.8136 grams (m_{Total}).

The chemist calculates the total number of tablets (n_{Calc}) by…

n_{Calc} = (241.8136 * 100) / (10.2121) = 2367.912 tablets

…and truncates the answer to report 2367 tablets **by weight**.

6.5 QC of Chemicals & Reagents, Standards and Controls

Purpose: This document outlines Quality Control (QC) procedures for Chemicals & Reagents, Standards and Controls to ensure, by routine testing of reagents and verification of standards, that when these materials are used in the performance of a technical procedure they will be of adequate quality.

Scope: All chemicals, reagents, standards and controls for use in the Forensic Chemistry Section, whether purchased or prepared in-house, will be subject to the QC procedures outlined in this document.

References:

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986.

1. **General Considerations**

   The quality of all chemicals purchased for use in the Forensic Chemistry Section will be adequate for their intended use. Generally this will mean that solvents, acids, bases, organic and inorganic compounds will be of ACS Reagent Grade or better.

   Water used for aqueous preparations should be distilled or deionized.

   Specific reagent formulations (which may be scaled up or down depending on need), QC procedures and lot documentation are located in the *Reagent Preparation Log*.

   Non-routine reagents that are prepared, will be subjected to QC testing appropriate for the reagent and recorded in the *Reagent Preparation Log*. The methods of preparation and verification must be recorded in the *Reagent Preparation Log*; otherwise all requirements are the same as those for routine reagents. However, non-routine reagents prepared for one time use may be recorded (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.

   Records regarding purchased standard verification are maintained in the 'Verification of Standards' section of the *Preparation Log for Controlled (or Non-Controlled Standards)*. The supporting documentation for the verification assertion is initially maintained in the *Verification Log for Controlled (or Non-Controlled) Standards*. An electronic version of this information will be maintained and updated at least

annually. The paper verification documentation will be archived in the Forensic Chemistry Section Chief's office. Documentation for prepared standards is maintained in the *Preparation Log for Controlled (or Non-Controlled) Standards*.

Standard designations are used for identity purposes for prepared standards. This designation is usually a five letter designation (i.e. ALPRA for alprazolam) followed by the last two digits of the current year it was prepared followed by a three digit number indicating how many times it has been prepared in the current year. Standard designations for prepared internal standards will use the same format (i.e. C13-08001). If a standard is prepared from a secondary standard, the standard designation will be followed by 'IH.'

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.

2. Chemicals & Reagents

A. Chemicals

i. Purchased Chemicals

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

Labeling: Original containers of chemicals will be marked upon initial opening by the opener's initials and the date of opening. Secondary containers to which chemicals are transferred should be marked with the content's identity and lot number.

ii. Prepared Chemicals

Preparation: 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.

B. Reagents

i. Purchased Reagents

Verification: Reagents will be purchased of appropriate quality and used as received.

Labeling: Containers of reagents will be marked upon initial opening by the opener's initials and the date of opening. Dispensers to which reagents are transferred should be marked with the reagent's identity and lot number.

ii. Prepared Reagents

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

Verification: Verification procedures for routinely prepared reagents are located in the *Reagent Preparation Log*. 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 Forensic Chemist. The verifier will initial the *Reagent Preparation Log* 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.

3. Standards

A. Purchased Standards

i. Controlled Substances

Verification: For each lot of a controlled drug standard that is purchased, an entry will be recorded in the Verification of Standards section of the *Preparation Log for Controlled Standards* which will include the lot number, method of verification, date and the verifier's initials (if applicable). Verification of controlled drug standards may be done by obtaining a Certificate of Analysis from the seller or by subjecting the material to testing by FTIR or GC/MS analysis by a Forensic Chemist.

If a Certificate of Analysis is not available, verification by direct analysis will be performed upon the first opening of the container and completed before

use of the standard in casework is permitted. Only one verification per lot number is required.

The verification documentation (Certificate of Analysis or spectral printouts) will be secured in the *Verification Log for Controlled Standards (see 6.5.1)*.

Labeling: Containers for controlled substance standards should be labeled initially with the date received and initials. When first opened the container should be labeled with the date of opening and the opener's initials.

ii. Non-controlled Substances

a. Drugs

Verification: For each lot of a non-controlled drug standard that is purchased, an entry will be recorded in the Verification of Standards section of the *Preparation Log for Non-Controlled Standards* which will include the lot number, method of verification and the verifier's initials (if applicable). Verification of non-controlled drug standards may be done by obtaining a Certificate of Analysis from the seller, or by subjecting the material to testing by FTIR or GC/MS analysis by a Forensic Chemist.

If a Certificate of Analysis is not available, verification by direct analysis will be performed upon the first opening of the container and completed before use of the standard in casework is permitted. Only one verification per lot number is required.

The verification documentation (Certificate of Analysis or spectral printouts) will be secured in the *Verification Log for Non-Controlled Standards (see 6.5.1)*.

Labeling: Containers for non-controlled substance standards should be labeled initially with the date received and initials. When first opened the container should be labeled with the date of opening and the opener's initials.

b. Internal Standards

Verification: Substances to be used as internal standards should be, as much as possible, consistently purchased from the same vendor and of the same purity. Should a change in vendor be necessary or desired, the internal standard stock solutions prepared from the new source should be

rigorously tested to ensure they perform as expected with historically established calibration curves.

Labeling: Containers of internal standard compounds should be labeled initially with the date received. When first opened the container should be labeled with the date of opening and the opener's initials.

c. Retail Products

Verification: Occasionally it may be necessary to compare case item(s) to a retail consumer product (non-drug). In these cases the product may be used as received provided there is no apparent damage to any anti-tampering devices.

Labeling: Containers for retail products should be labeled initially with the date received. When first opened the container should be labeled with the date of opening and the opener's initials.

B. Secondary Standards

While purchasing standard materials from legitimate vendors is preferred, it is recognized that situations exist which conflict with this preference. This may be due for example to a material's limited commercial availability and/ or excessive cost of the material. Consequently the Forensic Chemistry section may retain materials from casework or manufacture (by chemical syntheses, extraction(s) from plant or other materials, etc.) materials necessary for use in ***qualitative*** determinations by following the QC and documentation guidelines found in this section.

Verification: For each drug standard that is manufactured or retained from casework, an entry will be recorded in the Verification of Standards section of the *Preparation Log for Controlled (*or *Non-Controlled) Standards* which will include the secondary standard entry number (SS#), a unique identifier (i.e. the ASCL case number and item number if it was retained from casework), the method of verification, and the verifier's initials. Verification of secondary standards may be done by subjecting the material to testing by FTIR or GC/MS analysis by a forensic chemist.

For drug standards that are manufactured in-house, the method of preparation will be documented and included in the verification documentation. The method of preparation will include a listing of the

starting materials (with their lot numbers if applicable) and equipment used as well as a description of the actual procedure used in numbered sequence of steps. The documentation should be ***complete*** and ***understandable to others***!

Verification must be performed and completed before the standard is used in casework. The verification documentation (method of preparation, spectral printouts, etc.) will be secured in the *Verification Log for Controlled* (or *Non-Controlled*) *Standards*. (see 6.5.1)

Verification for standards retained from casework for secondary proficiency standards will be available in the electronic case file and is not to be entered in the *Verification Log for Controlled* (or *Non-Controlled*) *Standards* or the *Preparation Log for Controlled (or Non-Controlled) Standards.*

Labeling: Containers for secondary standards should be labeled with the identity of the drug, the secondary standard entry number and the unique identifier.

C. Prepared Standards

i. Standard Preparations Based on Purchased Materials or Secondary Standards
Verified purchased or secondary standards (controlled or non-controlled) may be manipulated to produce solutions containing a known amount (and/or identity) for use in quantitative[18] (and/or qualitative analysis) without subsequent re-verification. Internal standard solutions are treated separately (see Section 6.5 Part 3Biii).

The preparer should consult the Verification of Standards section of the appropriate *Preparation Log for Controlled* (or *Non-Controlled) Standards* to determine if the required verification has previously been performed on the lot of standard to be used. If this verification has not been performed, the Forensic Chemist must proceed with verification of the standard before using it in the preparation of any solution standards.

The method of preparation will vary depending on the type of analysis in which the standard will be employed. The Forensic Chemist is free to select an appropriate method of preparation, keeping in mind the intended use and

[18] Standards prepared from secondary standards may be used for ***qualitative*** analysis only.

the necessity of preparing the sample(s) for comparison in as identical a manner as possible.

The preparer should document the method of preparation in the Preparation of Standard Solutions section of the appropriate *Preparation Log for Controlled* (or *Non-Controlled) Standards*. The procedure description should be in enough detail so that another Forensic Chemist could produce a comparable standard solution.

Labeling: All prepared standards should be labeled with the standard's identity, standard designation, preparer's initials, the date of preparation, and the expiration date. Quantitative standards should also be marked with their concentration. All quantitative prepared standards expire one month after their preparation. Prepared qualitative standards expire one year after their preparation.

Standards prepared from secondary standards must also contain the designation "(IH)" following the standard designation.

ii. Internal Standard Solutions

Preparation: Specific formulations for internal standard stock solutions (IS) are contained in the *Internal Standard Preparation Log*. The actual amount of IS made, the lot numbers (if applicable) of all materials used, the standard designation and the preparer's initials will be recorded in the *Internal Standard Preparation Log*.

Verification:

Following the technical procedure for quantitations using an internal standard (see Section 6.6.5 Gas Chromatography), a quantitative check sample using a known amount of an appropriate drug standard (i.e. cocaine or codeine for C23 and methamphetamine or amphetamine or ephedrine/ pseudoephedrine for C13) will be prepared.

Run the check sample on a GC that has an established calibration curve for the drug standard used.

Compare the calculated amount of drug from the instrumental printout to the known amount of drug standard by performing a percent difference calculation.

EQUATION 2 ***Percent Difference***

$$\frac{(\text{Amount}_{known} - \text{Amount}_{calc})}{\text{Amount}_{known}} * 100$$

If the percent difference between the calculated and known amounts of drug is ≤ 5%, the internal standard stock solution is acceptable for use in casework.

If the percent difference between the calculated and known amounts of drug is > 5%, two additional independent quantitative check samples will be prepared.

- If both percent differences between the calculated and known amounts of drug are ≤ 5%, the internal standard stock solution is acceptable for use in casework.
- If one or both of the percent differences between the calculated and known amounts of drug are > 5%, the IS will be secured where it can not be used in casework. Additional steps should be undertaken to ascertain whether the problem is due to improper IS preparation or instrumental in nature.

Each new batch of IS that is prepared must be verified prior to use in casework. The verifier(s) will initial the *Internal Standard Preparation Log* for that batch of IS to certify that the calculated percent difference for the check sample fell within the acceptable tolerance range. Instrumental printouts documenting the IS verification will be secured in the *Check Sample Log* for the drug standard used.

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

4. Controls

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

6.6 Testing Techniques

6.6.1 Introduction

The following subsections of Section 6.6, describe the testing techniques commonly utilized for the analysis of evidence exhibits in the Forensic Chemistry Section. 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 these subsections should not be considered to exclude the use of other verified or published techniques. Chemists are encouraged to consult their peers and/or reference materials on an as needed basis.

The Forensic 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.

Deviations from these guidelines must have approval from the Forensic Chemistry Section Chief. These deviations must be documented and placed in the *SOP Deviations & Exceptions Binder*.

6.6.2 Color Testing

Purpose: The purpose of this document is to describe color tests routinely used by the Forensic Chemistry Section.

Reference(s):

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986.

U. S. Department of Justice, Drug Enforcement Administration (DEA). *Basic Training Program for Forensic Drug Chemists*.

ASCL, Forensic Chemistry Section. *Training Manual*.

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 and for determining the possibility of combining item contents within exhibits composed of multiple items.

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 in the Forensic Chemistry section 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 $NH_3(g)$:
      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 Duqenois-Levine
      This test is appropriate for testing vegetation or smoking device extracts for cannabinoids. Solvents normally used for the extraction include petroleum 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.
      g. Have another chemist verify the results. The chemist must initial and date that verification was performed in the analyst's 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. Heat the wire loop in the Bunsen burner flame until the wire begins to glow. Continue until no color is observed in the flame.
d. Dip the looped end of the wire into a sample[19]. 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.

[19] The sample may be solid or dissolved in a small amount of deionized water. If the sample is to be used in a solid form it may be helpful to dampen the wire loop with dilute hydrochloric acid before dipping it in the sample.

6.6.3 Thin Layer Chromatography (TLC)

Purpose: This document outlines the general method of analysis by Thin-Layer Chromatography (TLC).

Reference(s):

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986.

ASCL, Forensic Chemistry Section. *Training Manual*.

Dawson, N. *The Analysis of Anabolic Steroids Using a GC/FT-IR Technique*. Submitted as a poster presentation at the Southwestern Association of Forensic Scientists Meeting, 1990.

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 10 lists the most common solvent systems used in the Forensic Chemistry Section, however the use of any published TLC solvent system is acceptable.

TABLE 10 ***Common TLC Solvent Systems***

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 sample and standard spots are labeled uniquely. The chemist must be able to correlate the sample spot with the case and exhibit number.
iii. 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.
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 11 below.

TABLE 11 *Common TLC Plate Developing Techniques*		
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_2SO_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 a 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. Indicative Results

A spot on a TLC plate is indicative of a compound in the following instances:

i. A compound in a sample does not match the relative retention value and visualization reaction(s) of any standard on the same plate but the chemist suspects a particular compound based on past experience.

ii. A compound in a sample does match the visualization reaction(s) of a standard on the same plate but does not match the relative retention value of the standard by a small but significant change in retention value.

D. Negative Results

No spots 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 date of the testing,
iv. the standard designation of the standard used to make the identification (positive or indicative). (Exception: 6.1.3.4.C.i).

B. Positive Results

Any compound(s) meeting the criteria for positive results (as defined in 6.6.3 Part 4B) will be entered into the case notes by chemical name or an appropriate abbreviation.

C. Indicative Results

i. Any compound(s) meeting the criteria for indicative results (as defined in 6.6.3 Part 4C i) will be entered into the case notes by chemical name or an appropriate abbreviation followed by a question mark and the designation "NS" (i.e. No Standard) and the notation will be enclosed in parentheses.
ii. Any compound(s) meeting the criteria for indicative results (as defined in 6.6.3 Part 4C ii) 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 6.6.3 Part 4D) will be entered into the case notes by indicating negative results (i.e. "No spots", "Negative", "-", etc.).

If a compound in a sample does not match the relative retention value and visualization reaction(s) of any standard on the same plate and the chemist has no suspicion based on past experience of what the compound may be, it will be entered into the case notes by the number of unidentified spots. The chemist may include a drawing or description of spot colors and retention values relative to known standards.

6.6.4 Fourier Transform Infrared Spectroscopy (FTIR)

Purpose: This document outlines the general method of analysis by Fourier Transform Infrared Spectrometry (FTIR).

Reference(s):

Griffith, Peter R. and de Haseth, James A. *Fourier Transform Infrared Spectrometry*. John Wiley and Sons, Inc., New York, 1986.

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986

Mills III, Terry, Roberson, J. Conrad. *Instrumental Data for Drug Analysis (IDDA).* Elsevier Science Publishing Co., Inc., 1987.

Instrument Operation Manuals

ASCL, Forensic Chemistry Section. *Training Manual*.

U. S. Department of Justice, Drug Enforcement Administration (DEA). *Basic Training Program for Forensic Drug Chemists.*

Microgram

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 7.3.
2. **Instrument Operation Parameters**

TABLE 12 ***Routine Instrument Parameters for FTIR****	
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 Forensic 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).

Acceptable techniques:

- 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.

- Place cell over the top of the sample vial. Allow sufficient amount of time for vapor to enter the cell. Immediately close cell. Place cell 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

1. Common Sample Preparation Techniques

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

2. Running the Sample(s)

a. Solid Samples

A blank spectrum must be acquired by placing the anvil tip down against the crystal before each sample to ensure that both the anvil tip and crystal are clean. 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.

b. Vapor Samples

N/A in ATR.

c. Liquid Samples

A blank spectrum must be acquired by placing the anvil tip down against the crystal before each sample to ensure that both the anvil tip and crystal are clean. 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 check.

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.

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

i. A printout of the full original spectrum.
ii. A printout of the full spectrum that was subtracted.
iii. A printout of the subtraction results.

B. Positive Results

The sample's infrared spectrum[20] 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.). In addition, the blank spectrum does not contain a controlled substance or common cutting agent (positive or indicative match).

C. Indicative Results

An infrared spectrum[20] 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[21] 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.)
iv. The blank spectrum does not contain a controlled substance or common cutting agent (positive or indicative match).

D. Negative Results

[20] For vapor phase samples, only the region of 700cm-1 to 4000cm-1 should be considered.

[21] Exception: Peaks in the sample spectrum masked by other compounds may be excluded from this requirement if they are not the primary peaks used to distinguish between two compounds (i.e. cocaine base/HCl and pseudoephedrine/ephedrine).

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 instrumental printout(s).

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 Documentation

All spectral documentation, including blank spectrum, and its corresponding instrumental conditions must be stored electronically in the case file. Each spectrum must be labeled with the unique ASCL case number and exhibit number.

B. Positive Results

Any compound(s) meeting the criteria for positive results, as defined in 6.6.4 Part 5B, 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 6.6.4 Part 5C, 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 6.6.4 Part 5D, will be entered into the case notes indicating negative results (i.e. "No match", "Negative", "-", etc.) (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.)

6.6.5 Gas Chromatography (GC)

Purpose: This document outlines the general method of analysis by Gas Chromatography (GC).

Reference(s):
Grob, Robert L. *Modern Practice of Gas Chromatography*. John Wiley & Sons, Inc., New York, 1977.

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986.

ASCL, Forensic Chemistry Section. *Training Manual*.

U. S. Department of Justice, Drug Enforcement Administration (DEA). *Basic Training Program for Forensic Drug Chemists*.

Microgram

Scope: The methods in this document describe various techniques used to prepare samples and obtain gas chromatograms for qualitative and quantitative analysis, precautions and possible sources of error, data interpretation and notations specific to this test. This document is divided into the following categories:

1 Qualitative Analysis

A. Calibration

Refer to Section 7.4.

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 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 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 in the same manner 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 used to prepare the sample and standard.

ii. Running the Sample(s)

If the chemist has n samples for qualitative testing, blanks, standards and samples should be run by repeating the pattern bsBS in the following sequence:

$b_1 s_1 B_1 S_1 \ldots b_n s_n B_n S_n$
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 $b_n s_n B_n S_n$ must be run under identical chromatographic conditions.

If there is more than one analyte of interest in the sample, the sequence $b_n s_n B_1 S_{21} B_2 S_2$ may be followed.

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 in different solvents.
iii. Samples and standards do not have approximately the same concentration.

E. Data Interpretation

i. General

The data generated by the sequence $b_n s_n B_n S_n$ (or $b_n s_n B_1 S_1 B_2 S_2$) 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_n will only be compared with the t_R of S_n of the set)

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 13.

TABLE 13 ***Maximum Retention Time Match Tolerances***

Retention Time	**Tolerance**
≤ 3 minutes	± 2% relative
> 3 minutes	± 1% relative

The relative retention time is calculated according to EQUATION 6.6.5-1.

EQUATION 3 ***Relative Retention Time Calculation***

$$\frac{(t_{Rsample} - t_{RStandard})}{t_{RStandard}} * 100$$

ii. Positive Results

The calculated relative retention time of the sample (s_n) versus the standard (S_n) is ≤ the acceptable tolerance listed in TABLE 13.

iii. Indicative Results

N/A

iv. Negative Results

The calculated relative retention time of the sample (s_n) versus the standard (S_n) is greater than the acceptable tolerance listed in TABLE 13 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 spectra.

The relative retention time calculation(s) will be shown on the spectra 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/standard preparation,
- The standard designation of the standard,
- the results of the relative retention time calculation, and

b. Instrumental Documentation

All chromatograms (blanks, standards, and samples) must be stored electronically in the case file. Each spectrum must be labeled with the unique ASCL case number, exhibit number and date. 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 documentation).

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 Using Existing Calibration Curves

A. Calibration

Refer to Section 7.4.

B. Instrument Operation Parameters

Use TABLE 14 to select the GC method associated with the compound that is being quantitated. The method will set the instrument parameters.

TABLE 14 ***GC Methods for Existing Calibration Curves***

Compound(s) Quantitated	GC Method(s)[1] for GC#1	GC Method(s) for GC#2
methamphetamine	meth_frnt, meth_back	meth_frnt
amphetamine	amp_frnt	N/A
ephedrine or pseudoephedrine	eph_frnt, eph_back	eph_frnt
cocaine	coc_frnt, coc_back	coc_frnt
codeine	codeine	codeine

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

C. Technique

i. Sample Preparation

Use TABLE 15 to select the appropriate internal standard (IS) for the compounds listed.

TABLE 15 ***Internal Standard Information for Existing Calibration Curves***

Compound(s) Quantitated	IS[1]
methamphetamine	C13
amphetamine	C13
ephedrine or pseudoephedrine	C13
cocaine	C23
codeine	C23

[1] C13 = n-tridecane ($C_{13}H_{28}$), C23 = n-tricosane ($C_{23}H_{48}$)

After selecting the appropriate IS for the compound being quantitated, follow the steps listed in TABLE 16 (or TABLE 17 for Codeine) to correctly prepare the samples for quantitative analysis.

TABLE 16 *Sample Preparation for Existing Calibration Curves (except codeine)*	
❶	Weigh out a 10-100 mg portion[1] of the homogenized sample[2] to be quantitated and transfer to a screw-top vial.
❷	Record the amount in the case notes.
❸	Using a volumetric pipet and proper technique, transfer a 1 mL aliquot of the appropriate IS solution to the screw-top vial.
❹	Add ~2 mL of strong base solution[3].
❺	Add ~5 mL of methylene chloride.
❻	Cap the screw-top vial and vortex (~15 s) or shake (~1 min.)
❼	Centrifuge the screw-top vial and/or dry the methylene chloride layer over Na_2SO_4.
❽	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 ❶ (see below, Section 6.6.5 Part 2Cii). [3] Normally 10% KOH, NaOH, etc.	

TABLE 17 *Sample Preparation for Existing Calibration Curves-Codeine*	
❶	Using a volumetric pipet and proper technique, transfer a 5 mL aliquot of the sample[1] to a 25mL volumetric flask.
❷	[2] Dilute the sample in the volumetric flask to 25mL with H_2O and mix well by shaking.
❸	Using a volumetric pipet and proper technique, transfer a 5 mL aliquot of the sample solution to a separatory funnel.
❹	Using a volumetric pipet and proper technique, transfer a 1 mL aliquot of C23 solution to the separatory funnel.
❺	Add enough weak base solution[3] to make sample solution basic.
❻	Add ~5 mL of methylene chloride.
❼	Cap the separatory funnel and shake/vent until solution is mixed well.
❽	Collect the methylene chloride layer and dry over Na_2SO_4.
❾	Transfer a portion of the methylene chloride layer to an auto-sampler vial and cap.

[1] 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 ❶ (see below, Section 6.6.5 Part 2Cii). [2] For extremely weak samples, this dilution step may be omitted and/or a larger aliquot tested. [3] Normally, saturated $NaHCO_3$ solution.

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[22] the percent difference between the known amount of standard in the check sample versus that calculated from the calibration curve using EQUATION 4.

EQUATION 4 ***Percent Difference***

$$\frac{(Amount_{known} - Amount_{calc})}{Amount_{known}} * 100$$

If the percent difference is ≤ 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 method.

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 [**NOTE:** triacetin is known to co-elute with C13].
v. The wrong IS (or no IS) is transferred to the screw-top vial.

[22] For codeine, the calculated compound amount generated by the Chemstation software is 'mg/mL.' Because the check sample is a 5mL aliquot of a 25mL standard dilution, the number generated by the software must be multiplied by 5mL to determine the amount of codeine present.

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).

The percent purity is calculated according to EQUATION 5.

EQUATION 5 ***Percent Purity***

$$\frac{\text{Amount}_{calc}}{\text{Amount}_{known}} * 100$$

The calculated compound amounts on the GC generated report are reported ***as the hydrochloride salt***[23]. It may also be necessary for the chemist to convert their answer to the base or a different salt form. See TABLE 18 for some common conversions.

TABLE 18 ***Common Salt/Base Conversions***

Compound	**Conversion**	**Factor**
Amphetamine	Hydrochloride to Base	0.7876
	Sulfate to Base	0.7378
	Sulfate to Hydrochloride	0.9368
Cocaine	Hydrochloride to Base	0.8927
Codeine	Phosphate to Base	0.7533
Heroin	Hydrochloride to Base	0.8714
Methamphetamine	Hydrochloride to Base	0.8036

ii. Positive Results

N/A

iii. Indicative Results

N/A

iv. Negative Results

N/A

F. Notations Specific to This Test

[23] For codeine, the calculated compound amount on the GC generated report is reported ***as the phosphate salt***.

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 spectra.

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

a. Case Notes

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

- Type of sample preparation
- standard designation of IS used to prepare sample
- the sample amount(s) used,
- the calculated percent purity, and

b. Instrumental Documentation

All chromatograms (blanks, standards, and samples) must be stored electronically in the case file. Each spectrum must be labeled with the unique ASCL case number, exhibit number and date. Positive Results

N/A

ii. Indicative Results

N/A

iii. Negative Results

N/A

3 Quantitative Analysis by Creating a New Calibration Curve

Theory:

For compounds which do not have a pre-prepared calibration curve, the chemist will make a three (3) point calibration curve using the internal standard method. A carefully measured quantity of an internal standard substance is introduced into each standard and sample, and the ratio (S_a/S_r) of analyte peak area (S_a) to internal standard peak area (S_r) is the analytical parameter.

The concentrations of the standard samples should be chosen so that the unknown sample concentration will fall within the standard concentration range. The internal standard should be chosen based on the characteristics of the sample. The retention time of the internal standard must not correspond to the retention time of any component of the sample, and the retention time of the internal standard should be comparable to the retention time of the analyte.

A calibration curve is prepared in which this ratio (S_a/S_r) is plotted versus analyte concentration of the standards. The S_a/S_r value for the sample is used to determine the analyte concentration in the sample. A MS Excel spreadsheet is available which will perform the calculations after the chemist has entered the measured peak areas.

A. Calibration
   Refer to Section 7.4.

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 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 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.), and 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 chemist will prepare…

a. Three standard solutions, each of a different concentration, which include identical amounts of the chosen internal standard. [**NOTE:** The three standards should be prepared independently and not through serial dilution.]
b. One (or more) sample solutions which contains an amount of the chosen internal standard identical to that in the standards.
c. One sample solution, prepared in a manner identical to the solutions in "a" & "b", but which ***contains no internal standard***.
d. One internal standard solution, prepared in a manner identical to the solutions in "a" & "b", but which ***contains no analyte***.
e. A solvent blank.

The chemist will prepare the necessary solutions according to the steps in TABLE 19, ***being careful to detail the specifics of their procedure in the case notes***.

TABLE 19 *Sample Preparation For Creating a Calibration Curve*
❶ Weigh out three different amounts of the known standard, and a 10-100 mg portion of the sample[1] to be quantitated. Transfer each weighing to a separate screw-top vial.
❷ Record the amounts in the case notes.
❸ Transfer an identical amount of the internal standard into each of the tubes.
❹ Depending on the compound to be quantitated, a solvent dilution, or an acid/base extraction may be performed.
❺ Cap the screw-top vial and vortex (~15 s) or shake (~1 min.)
❻ Centrifuge the screw-top vial. The organic layer may also be filtered and/or dried (over Na_2SO_4) at this point if appropriate.
❼ Transfer a portion of the organic layer to an auto-sampler vial and cap.
[1] A larger portion (i.e. > 100 mg) may be necessary for extremely weak samples.

ii. Running the Sample(s)

A solvent blank will be run before each standard and sample using the same GC method.

Blank, standard and sample aliquots are introduced into the GC by automated injection. A report, showing the chromatogram and any measured peak area(s), should be generated at the conclusion of the method.

D. Precautions & Possible Sources of Error

i. Not using identical amounts of internal standard in the standards and samples.

ii. Co-eluting peaks.

E. Data Interpretation

i. General

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

The chromatograms of the sample solution containing no internal standard and the internal standard solution containing no sample must prove that the retention time of the internal standard does not correspond to the retention time of any component of the sample.

The chemist will demonstrate the reliability of the prepared standards, and the linearity of the calibration curve that they represent, by showing that the coefficient of determination (r^2), derived by calculating the linear regression equation based on the measured data and the known concentrations, is ≥ 0.999. A MS Excel spreadsheet is available to perform the calculations after the chemist has entered the measured peak areas.

The calculated compound amount and purity from the spreadsheet calculations are shown ***as the same salt (or base) form as the standard used by the chemist***. It may also be necessary for the chemist to convert their answer to a different form of the compound.

ii. Positive Results

N/A

iii. Indicative Results

N/A

iv. Negative Results

N/A

F. Notations Specific to This Test

i. General

The chemist must describe the specifics of their procedure in the case notes.

a. Case Notes

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

- Type of sample preparation,
- the standard designation of the IS used,
- the standard and sample amount(s) used,
- the calculated percent purity, and

b. Instrumental Documentation

All chromatograms (blanks, standards, and samples) must be stored electronically in the case file. Each spectrum must be labeled with the unique ASCL case number, exhibit number and date. 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 documentation).

ii. Positive Results

N/A

iii. Indicative Results

N/A

iv. Negative Results

N/A

6.6.6 Gas Chromatography/Mass Spectrometry (GCMS)

Purpose: This document outlines the general methods of analysis by Gas Chromatography/Mass Spectrometry (GC/MS).

Reference(s):

McLafferty, F.W. *Interpretation of Mass Spectra*. University Science Books, Mill Valley, CA, 1980.

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986.

U. S. Department of Justice, Drug Enforcement Administration (DEA). *Basic Training Program for Forensic Drug Chemists*.

ASCL, Forensic Chemistry Section. *Training Manual*.

Microgram

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

1. **Calibration & Maintenance**
   Refer to Section 7.5.

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/blank 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.
Low sample concentration.

C. Running the Sample(s)
Co-eluting compounds.
Scan range may have to be adjusted.

5. Data Interpretation

A. General

Identification of unknown compound(s) in a sample is based on comparing the sample's mass 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 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.
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[24] is ≥ 10.
ii. The identified compound is not present in the preceding solvent blank and the preceding blank does not contain any peaks (signal to noise ≥ 3) whose mass spectrum is a positive or indicative match for a controlled substance or a common cutting agent.
iii. Both the sample and reference spectra must show the molecular ion peak if it is commonly attainable for a particular substance.
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).

[24] It is recommended that a fully resolved peak is present in the chromatogram.

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 Forensic Chemist will include in the case notes, for each GC/MS test performed, the following information:
   a. Type of sample preparation

ii. Instrumental Documentation

All chromatograms (blanks, standards, and samples) must be stored electronically in the case file. Each chromatogram/spectrum must be labeled with the unique ASCL case number, exhibit number and date. 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 documentation).

B. Positive Results

Any compound(s) meeting the criteria for positive results, as defined in 6.6.6 Part 5B, may be entered into the case notes by chemical name or an appropriate abbreviation (Note: Controlled substances must be entered into the case notes).

C. Indicative Results

Any compound(s) meeting the criteria for indicative results, as defined in 6.6.6 Part 5C, 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 (Note: Controlled substances must be entered into the case notes).

D. Negative Results

i. Any compound(s) meeting the criteria for negative results, as defined in 6.6.6 Part 5D, are generally not included in the notes.

ii. If the chromatogram contains no peaks, the results will be entered into the case notes indicating negative results (i.e. no peaks, negative, etc.) If the mass spectra of all peaks in the sample's chromatogram do not visually match any available reference standard spectra, the results will be entered into the case notes indicating negative results (i.e. no match, no ID, negative, 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.)

6.6.7 X-Ray Fluorescence

Purpose: This document outlines the general method of analysis by X-ray Fluorescence Spectrometry (XRF).

Reference(s):
Shimadzu Energy Dispersion Fluorescence X-ray Spectrometer Instruction Manual.

ASCL, Forensic Chemistry Section. *Training Manual*.

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.

1. **Calibration & Maintenance**
   Refer to Section 7.6.

2. **Instrument Operation Parameters**

Table 20 ***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.
         b. ***If the sample is a liquid:*** The sample is spotted onto a piece of wiping paper or filter paper.

[**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.]

iii. A second piece of disposable thin film is placed on 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 if a vacuum is required.

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 adjust operating to obtain the best possible results.

4. Precautions & Possible Sources of Error

- X-ray fluorescence cannot detect any element below Sodium in the periodic table. Nitrogen, oxygen, fluorine, and neon can only be seen under special conditions and thru special preparations.
- XRF cannot determine the oxidation state of the element.
- Contamination of the sample compartment.
- 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 known XRF emission energies of the element and the following criteria are met:

i. All major emission lines[25] for the element are present (i.e. proper energies and patterns) in the sample.

[25] Iodine must have at least three resolved peaks, two of which exceed 0.100 cps/μA net intensity. For other elements the primary resolved peak must have a net intensity of at least 0.100 cps/μA.

ii. All emission line intensities in the sample are in the proper ratios compared to the element (Exception: If another element's emission line(s) are overlapping with the element of interest emission line(s), these line(s) may be omitted from ratio comparison. The interfering emission line must be labeled in addition to the element of interest.)

iii. The sample must have a net intensity of 0.100 cps/μA for the element of interest[25].

C. Indicative Results

The sample's XRF spectrum is visually similar to that of the known XRF emission energies of the element, but the sample's spectrum does not meet all of the criteria for a positive result.

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)

ii. Instrumental Printouts

All spectra and their corresponding instrumental conditions must be stored electronically in the case file. Each spectrum and all corresponding data must be labeled with the unique ASCL case number, exhibit number, and date.

B. Positive Results

Any compound(s) meeting the criteria for positive results, as defined in Section 6.6.7 Part 5B, will be entered into the case notes by the element name or an appropriate abbreviation.

C. Indicative Results

Any compound(s) meeting the criteria for indicative results, as defined in Section 6.6.7 Part 5C, will be entered into the case notes by the element 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 6.6.7 Part 5D, will be entered into the case notes by indicating negative results (i.e. “No match”, “Negative”, “-“, etc.).

6.6.8 Pharmaceutical Identifier

Purpose: This document outlines general methods used for the visual identification of pharmaceuticals.

Reference(s):

Drug Identification Bible. Amera-Chem, Inc., Grand Junction, CO.

Johnson, J. and Chapman, K. *The Med-Scan Manual*, Comprehensive Drug ID Source. Med-Scan International, Inc.

Physician's Desk Reference. Medical Economics Company, Inc., Montvale.

Physician's Desk Reference Generics. Medical Economics Company, Inc., Montvale.

Physician's Desk Reference for Nonprescription Drugs. Medical Economics Company, Inc., Montvale.

The Logo Index for Tables and Capsules. Drug Enforcement Administration Office of Forensic Sciences Special Testing and Research Laboratory.

Arkansas Poison Control Center (1-888-228-1233 or 501-686-5072), University of Arkansas Medical Sciences Pharmaceutical Building, Little Rock.

http://www.rxlist.com

Scope: The methods described in this document can be used to aid in the identification of pharmaceuticals, in the form of tablets and capsules, submitted for drug analysis. Other references not listed may also be used[26].

1. **General Method**
   - Record the physical appearance of the tablet/capsule in the case notes (see 6.6.8 Part 4A).
   - Compare sample characteristics/appearance to reference source(s).

[26] Manufacturer labels or other markings on sealed blister packs, bottles and ampules are approved references for pharmaceutically identifying a container's contents.

2. Precautions & Possible Sources of Error

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

3. 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

The active ingredient(s) of the tablet/capsule could not be identified because of one or more of the following:

i. All markings on the sample were not visible[27] (e.g. broken or partial tablets or capsules)

ii. All appearance description(s) do not match those of reference source(s) (e.g. the tablet does not contain a score, but the reference material indicates that a score is present).

iii. Conflicting identifications are obtained from different reference sources.

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.

4. 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

[27] Exception: In an exhibit item that contains whole and partial tablet(s), if the partial tablet(s) contain similar markings as the whole tablets, they can be positively identified with the whole tablets.

The active ingredient(s) of any tablet/capsule identifications meeting the criteria for positive results, as defined in Section 6.6.8 Part 3B, 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 6.6.8 Part 3C, 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 6.6.8 Part 3D.

6.7 Reporting of Analytical Results

An ASCL "Report of Laboratory Analysis" is generated at the conclusion of analytical testing. For the Forensic Chemistry Section these reports normally consist of "header" information, a list of "Items" and the "Test Results" for each item listed.

This document describes general guidelines intended to cover reporting of the majority of cases analyzed by the Forensic Chemistry Section. However situations may occur requiring deviation from these guidelines due to the extreme variability of evidence received. In such a case, the chemist will consult the Section Chief to determine an approved method for reporting the information.

1. Guidelines

A. All items (exhibits) documented in the chemist's case notes, including items not tested, should be included on the report. Consecutive items with the same test results may be combined for reporting.

B. For items (exhibits) that ***were not tested*** the report should include:
   i. The item (exhibit) number.
   ii. "not tested" listed under Test Results.
   iii. Items excluded from testing in controlled substance manufacturing cases may be listed as a group at the end of the report.

C. For items (exhibits) that ***were tested*** the report should include:
   i. The item (exhibit) number.
   ii. One or more values of initial exhibit amount[28], as recorded in the case notes, with units.
      a. If the substance was less than an amount that can be weighed on the analytical balance, it will be listed as a residue on the report if a visible substance is present.
      b. Weights[29] should be listed in grams or kilograms.
      c. Volume should be listed in milliliters or liters.
      d. Tablets/capsules/pharmaceutical patches (see Table 8).
      e. Calculated tablet/capsule counts should be reported as "x tablets (or capsules) by weight."
      f. Exhibits that tested positive for LSD (see Table 8).

[28] Initial exhibit amount may be omitted for evidence in manufacturing cases if a representative sample was taken by the analyst in the field.

[29] Wet solids may be excluded from the measurement requirement with approval of the supervisor.

iii. The identities of elements, compounds or substances that were positively identified. Only elements, compounds or substances meeting the criteria of Section 6.2 Parts 1C & 1D may be reported.

a. For compounds meeting the above criteria, a salt/base form or stereoisomer may be reported based on a positive or indicative IR spectrum.

b. For tablets/capsules pharmaceutically identified to contain a combination of controlled and non-controlled active ingredients and…
   - one or more of the non-controlled ingredient(s) does not meet the minimum testing requirement for reporting[30] and
   - all controlled ingredients have been positively identified[31],

   the results may be reported by listing the controlled and non-controlled ingredients positively identified by testing followed by the phrase "identified* as" and a list of all pharmaceutically identified controlled and non-controlled ingredient(s)[32].

c. For tablet(s)/capsule(s) that have been pharmaceutically identified and positively identified[30] to contain pseudoephedrine (or ephedrine), the results may be report by listing pseudoephedrine (or ephedrine) followed by the phrase "identified* as" and pseudoephedrine (or ephedrine) with the salt/base form and dosage[33].

d. All results reported with the terminology "identified* as" shall 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."

e. If no element, compound or substance was positively identified, the results may be reported as "no controlled substances detected."

f. If only elements or non-controlled substances were positively identified in an exhibit (if pharmaceutically identified see 6.7.1.C.iii.d), the chemist may

[30] i.e. one positive category "A" test.

[31] i.e. one positive category "A" test and one positive category "B" test or two positive category "A" tests per compound.

[32] e.g. a tablet is pharmaceutically identified to contain hydrocodone and acetaminophen. The sample is extracted prior to mass spectroscopy in such a way that most of the acetaminophen is removed and therefore only the hydrocodone is positively identified by testing. The chemist shall report the results as "hydrocodone" with "identified* as acetaminophen, hydrocodone" as a separate entry.

[33] e.g. a tablet is pharmaceutically identified to contain pseudoephedrine HCl 30mg/ tablet. The chemist shall report the results as "pseudoephedrine" with "identified* as pseudoephedrine HCl 30mg/ tablet" in a separate entry.

g. 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.
h. Use TABLE 21 to determine the proper method to report results which may be significant in manufacturing cases.

TABLE 21: *Reporting Results Significant to Manufacturing Charges*

Result Reported	Necessary Tests
Phosphorus/Iodine	XRF
Ammonium Nitrate	IR[4] solid
Ammonia[1]	IR[4] vapor, Nessler's
Lithium[2]	IR[4] solid, Flame Test, XRF
Lithium metal	IR[4] solid, Flame Test, XRF, Reactive w/ H_2O
Sodium[3]	XRF, IR[4] solid
Sodium metal	XRF, Reactive with H_2O
Sodium Nitrate	IR[4] solid

[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").

iv. Quantitative results (if applicable).

Quantitative results may be reported as:

a. A percentage (normal method for solids). Truncate (do not round) the calculated percent purity to three (3) significant figures with the following exceptions:
   - If the calculated percent purity exceeds 99%, report the purity as 99%.
   - If the calculated percent purity is below 1%, report the purity as "less than 1%."

b. A concentration (normal method for liquids or tablets) with the correct number of significant figures and truncated.
c. The total amount of compound.

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 header to match the information on the *Evidence Submission Form*.

B. Analytical Results

The chemist should compare the items and test results on the report to those 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

When analytical conclusions and/or opinions are made on evidence submitted for analysis, the chemist will proofread and electronically sign their report ensuring the report is accurate and error-free. A report will be issued to the investigating agency.

3. Supplemental/Amended Reporting

A. Supplemental Report

If additional evidence is received or additional requests for analysis are made *after* the original report has been issued, a supplemental request must be made. Once the evidence is analyzed, a supplemental report must be issued.

When a supplemental report is issued, the words "Supplemental Report" must appear below the header information and above the listing of the evidence and the results (Note: The request in Justice Trax must be a 'supplemental drug request' in order for "Supplemental Report" to be generated on the laboratory report).

If the supplemental request requires a quantitation from previously reported qualitative results, the initial weight/volume from the original report must be reported on the supplemental report[34].

Note: When additional evidence is received on a case that has not been completed, the additional evidence may be analyzed and included on the original report.

B. Amended Report

An amended report is necessary if an error is found on the original report.

If additional analysis is required after the original report has been issued, an amended request must be made. The evidence must be analyzed and an amended report issued.

When an amended report is issued, the words "Amended Report" must appear below the header information and above the listing of the evidence and the results (Note: The request in Justice Trax must be an 'amended drug request' in order for "Amended Report" to be generated on the laboratory report).

[34] If there is a substantial difference between the original reserve weight and the supplemental initial weight (i.e. the substance has gained/lost weight because of water), the supervisor must be notified to determine the weight to be reported.

7. Calibration and Maintenance

7.1 Introduction

This section describes the basic calibration and maintenance procedures used in the Forensic Chemistry Section.

Any instrument that is not in proper working condition is "Out of Service" and can not be used for casework. These instruments must be clearly labeled so they will not be used for casework.

Instruments that fail their calibration must be clearly labeled as "Out of Service". After proper maintenance has been performed and the instrument passes all the calibration tests, the instrument is returned to service for casework.

Instruments that do not have calibration procedures are "Out of Service" until calibration procedures are validated and approved by the Chief Forensic Chemist. The procedures must be added to the Forensic Chemistry Quality Manual before the instrument is used for casework.

7.2 Balances

Purpose: To provide a guideline for the proper maintenance and calibration of the balances in the Forensic Chemistry Section.

Calibration & Maintenance

An *Analytical* or *Toploading Balance Performance Verification Log Sheet* is provided to each Forensic Chemist for each balance they are issued for use. Each Forensic Chemist's balance(s) will be subjected to the checks in TABLE 22 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 22 *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 verified?	Weigh and record verification weight
Was the balance within tolerance? 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	If no, Calibrate before use ☹ If yes, Balance ready to use ☺

In addition to the daily use checks, the Forensic Chemist will subject their balance(s) to the checks in TABLE 23 on a monthly basis. The results of these checks and the serial number of the calibrated weight (or identifying number) used for the checks will also be recorded on the appropriate log sheet.

TABLE 23 *Required Monthly Balance Checks*	
Monthly Checks	**Actions**
Has the balance been verified with two other weights (e.g. 1g and 20 g for top-loading balance)?	Weigh and record the weights
Was the balance within tolerance? Analytical balance – 1 g ± 0.2 mg Analytical balance – 200 mg ± 0.1 mg Toploader balance – 20 g ± 0.00 g, 2000 g ± 0.00 g Bulk balance (bulk room) – 4000 g ± 0.00 g, 25 lb. ± 0.00 lb. Bulk balance (evidence) – 20 kg ± 0.00 kg, 50 kg ± 0.00 kg	If no, Calibrate before use ☹ If yes, Balance is ready to use ☺

If the balance will not calibrate or if it is not in tolerance after it has been calibrated, the balance must be removed from service for repair. After the balance has been repaired, the balance must be leveled and calibrated before it is returned to service. All repairs, maintenance, and standard weights used must be documented on the appropriate log sheet.

If the Forensic 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 verification has been performed that day. If the balance has not been verified, the required 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.

7.3 Infrared Instruments

Purpose: To provide a guideline for the proper maintenance and verification of the FTIRs in the Forensic Chemistry Section.

1. Calibration

The verification of each FTIR must be checked monthly and after any maintenance has been performed. The following steps should be performed to check the calibration:

Nicolet 360 FTIR(s) without ATR Accessory:

1. Clean the sample compartment.
2. Wait 30 minutes before continuing.
3. Scan a new background.
4. Place polystyrene sample card into the sample compartment.
5. Run the macro POLYQC.MAC.
6. Assess the verification report for the pass/fail requirements.
7. Index the verification report to the designated ASCL case # for the instrument
8. Record the results on the FTIR Avatar Log Sheet.
9. If the instrument passed all checks, it is ready to use.
10. If the instrument failed any of these checks, the instrument must be removed from service for repair.

Nicolet 380 FTIR(s) with ATR Accessory:
Be gentle with the ATR accessory!!!

1. Remove the purged air connection from the instrument.
2. Remove the ATR accessory.
3. Place transmission plate in the FTIR (a screen should appear indicating 'transmission experiment setup') and place the FTIR cover on instrument.
4. Align the bench (>Collect>Experiment Setup>Diagnostic>Align).
5. Run the Valpro Qualification (>Analyze>Valpro Qualification>Nicolet 380 System KBr-EP).
6. 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.*
7. 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'.).

8. 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 (It is recommended to turn the anvil until it 'clicks' three times to ensure equal pressure).
9. 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*.
10. 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.
11. If the instrument failed any of these tests, the instrument must be removed from service for repair.

2. Maintenance

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

7.4 Gas Chromatographs

Purpose: To provide a guideline for the proper maintenance and calibration of the GCs in the Forensic Chemistry Section.

Calibration & Maintenance

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 24 *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 25 *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 ≤ 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.

7.5 Gas Chromatographs/Mass Spectrometers

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

1. Calibration

Each GC/MS should be calibrated daily (before use) using the Autotune feature of the Chemstation software. The Autotune uses PFTBA (Perfluorotributylamine) masses 69, 219, and 502 to optimize various parameters for the Mass Selective Detector (MSD). A calibration report is generated (FIG. 1).

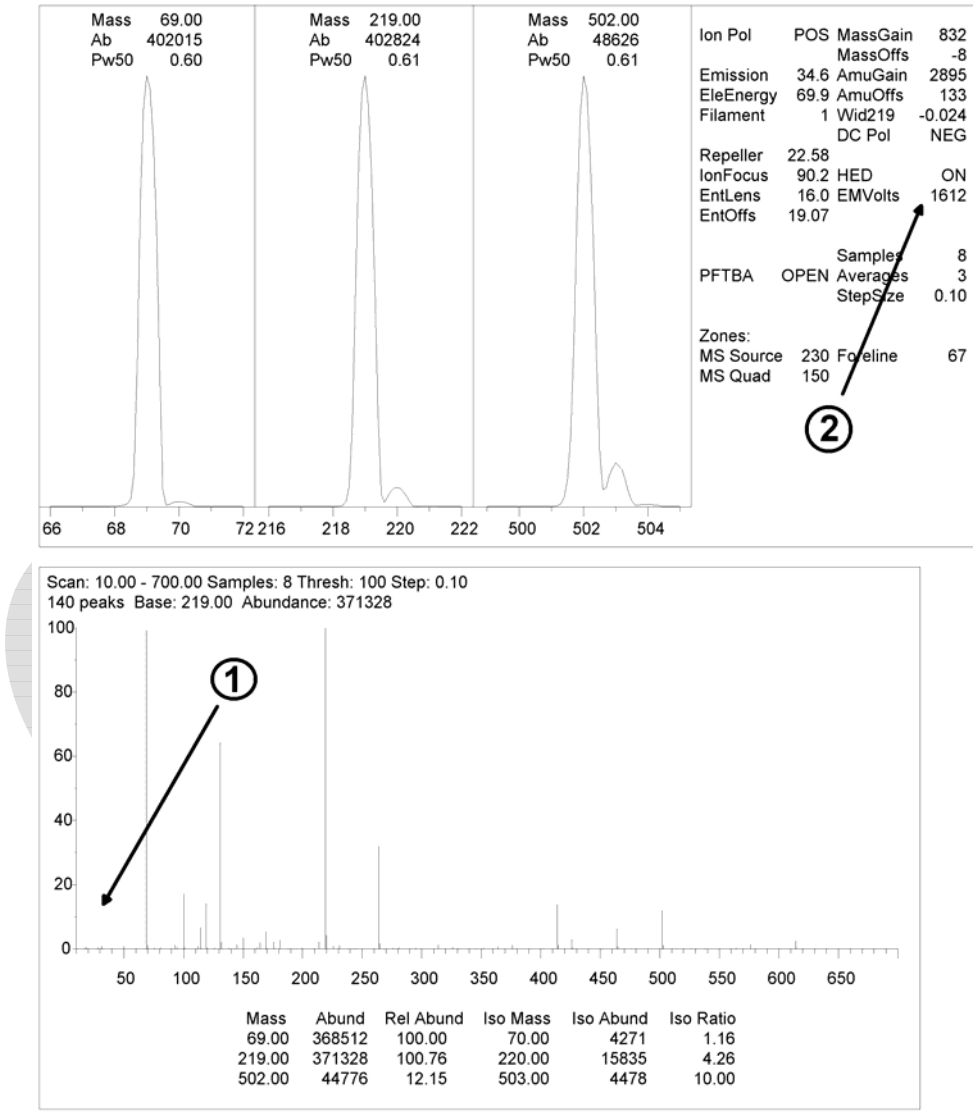

FIG. 1 *Important areas of the MSD Autotune report.*

The chemist must assess the calibration by examining the labeled areas of the Autotune report (FIG. 7.5-1①,②) for the following conditions:

①Examine the 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,
②and determine whether the EM voltage is > 2900.

If any of these conditions exist, the instrument is not in proper working condition and should be removed from service until it has been repaired and has passed calibration.

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

2. Routine Maintenance

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 calibration was performed and when filaments have blown.

TABLE 26 *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 should calibrate 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 27 *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

7.6 X-Ray Fluorescence Spectrophotometer (XRF)

Purpose: To provide a guideline for the proper care and maintenance of the X-ray fluorescence instrument.

1. **Verification**

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

   Fe 68.5 – 73.0%
   Cr 16.0 – 20.5%
   Ni 6.0 – 10.5%
   Mn 0.5 – 3.5%
   Mo 0.05 – 0.45%
   Energy for Fe: 6.40 ± 0.05 keV
   Resolution for Fe: <165eV

   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**

   This instrument requires the detector to be cooled by liquid nitrogen. The Dewar for this instrument should be filled a minimum of every third day, when practical. 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 a calibration using a disk composed of aluminum and copper, and the EDX software (under "instrument calibration").

8. Proficiency Testing Program

Each chemist in the Forensic Chemistry Section participates in a proficiency testing program. This program includes…

I. A minimum of one chemist 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.
- Internal proficiency tests may include previous external proficiency samples, samples retained from casework (secondary proficiency standards), samples made from primary standards, re-examination techniques, and blind techniques.

8.1 Evaluation of Results Obtained

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

Qualitative Analysis		
	Pass	**Fail**
Controlled substance(s)	Correct identification of all controlled substances present.	Incorrect or incomplete identification.
Non-controlled substances	N/A	Incorrect identification.

Quantitative Analysis (if required)		
	Pass	**Fail**
Controlled substance(s)	Results ≤ 5% relative difference or ≤ 1% absolute difference.	Results > 5% relative difference and > 1% absolute difference.
Non-controlled substances	N/A	Results > 5% relative difference and > 1% absolute difference.

9. Case Records

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 considered the official case record.

9.1 Technical & Administrative Review

All cases will be technically and administratively reviewed. The review process must confirm that electronic versions of all necessary documentation are in the imaging module of the LIMS-plus program.

9.2 Mistakes

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 Section Chief (or designee) for resolution.

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

10. Miscellaneous

10.1 Inspection of Illicit Laboratory Evidence Samples

Purpose: This document outlines the general method for rendering illicit laboratory samples safe prior to submission to the Arkansas State Crime Laboratory.

Reference(s):
Greenwood, N. N., Earnshaw, A. *Chemistry of the Elements*. Pergamon Press, Tarrytown, NY, 1993.

Scope: The methods in this document describe the protocol for assessing and ultimately rendering safe illicit laboratory samples that law enforcement agencies have sampled themselves and are submitting to the Arkansas State Crime Laboratory for analysis. Also included in this document are common chemical categories submitted to the Arkansas State Crime Laboratory and the safety hazards and precautions associated with those chemical categories.

1. 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 section member should be called. The chemist responsible for rendering samples safe has the authority to refuse any samples that are deemed unsafe for storage.

A. 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.

B. 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 Illicit Laboratory chemist.

2. Packaging of Evidence

A. 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. 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.

B. 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.

C. 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.

D. 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.

E. 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.

F. Packaging of Sharps

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

G. 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 such as but not exclusive to tuberculosis, hepatitis C, anthrax, or the "flesh eating" virus.

3. 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.

4. 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, an illicit laboratory safety sheet may not be tendered to properly submit the evidence.