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<td><strong>New:</strong> SD-03-06, SD-06-08</td>
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**NOTE:** When revised, chapter numbers will be listed in this table as they appear on the effective date, with former chapter numbers shown in parentheses.
01 REPORTING GUIDELINES AND DOCUMENTATION

SD-01-01 REPORTING GUIDELINES

1 Scope
To establish standards for reporting the results from the analysis of seized drug evidence examined by chemists in the DPS Crime Laboratory System.

2 Reporting Guidelines for Analytical Results
DPS reporting guidelines are based on the laws and definitions provided in Chapters 481-485 of the Texas Health and Safety Code (HSC) which contains the Texas Controlled Substances Act. The law determines the terminology used in reporting the identification of most controlled substances and requires the weight of that substance to establish the penalty group.

2.1 Reporting Results of Controlled Substances and Dangerous Drugs

A. General Reporting Examples of Identification

1. Report the identification of a controlled substance as it appears in the Texas Controlled Substances Act.

2. Precede the name of all substances identified with the word “Contains”.

3. The salt form or base form may be reported if it has been properly identified.

4. If a controlled substance(s) and a dangerous drug(s) are identified in a sample, the analyst should normally report only the controlled substance(s). At the discretion of the analyst, it may be necessary to report other substances identified.

5. If pharmaceutical identification is used to identify an isomer, then a footnote must be applied, such as:

   “Information from the pharmaceutical company indicates the dextrorotatory isomer.”

6. Steroid esters may be reported by either the steroid alcohol name or by the steroid ester name, if so identified.

   Examples: Contains Testosterone or Contains Testosterone Cypionate

7. If a sample is examined for the presence of a volatile chemical, e.g. as defined in HSC 485, and one is identified, report the results.

8. If identifying a dangerous drug, controlled substance, or non-controlled substance that has a trade name, report it by its common generic name.

   Example: Contains Oxycodone (not OxyContin)

9. Reporting controlled substances in pharmaceutical preparations which may be placed into multiple penalty groups

   a) For items such as tablets or capsules which can be determined to be virtually identical and there is no reason to suspect tampering or counterfeiting, and for preparations in sealed pharmaceutical containers or packages, report the controlled substance identified and the following footnote:

   “Pharmaceutical identification indicates…”

Printed copy is uncontrolled. Refer to electronic copy for current version.
b) For preparations in containers and packages not possessing a manufacturer’s seal or identifier where other non-narcotic ingredient(s) are present:
   i. Report the identity of the controlled substance and attempt to also confirm and report the non-narcotic ingredient. An appropriate footnote may be added, such as:
      “Contains associated non-narcotic ingredient(s) commonly found in a pharmaceutical preparation.”
   ii. If the analysis indicates non-narcotic ingredient(s) but is unable to be confirmed, report only the controlled substance with the accompanying footnote:
      “Non-narcotic ingredient(s) commonly found in pharmaceutical preparation were indicated, but not confirmed.”

B. Reporting Marihuana, Cannabis sativa L., and THC
   1. For reporting Marihuana and Cannabis sativa L., refer to SD-03-06.
   2. If a significant amount of an impurity, such as tobacco, is present in the plant sample, a conservative visual or microscopic estimate of the percent of plant present may be documented in the case record.
   3. If insufficient physical characteristics are present to identify marihuana, but THC has been identified, report as “Contains Tetrahydrocannabinol(s).”
   4. Suspected cannabis seeds will not be examined. Report as “No Analysis.”

C. Reporting Chemicals that are Controlled Based on Structural Classification
   1. Report the classification of the identified substance(s) as listed in the HSC.
   2. The name of the controlled substance will be displayed on the report along with the legal classification and an accompanying footnote that specifies the section(s) of HSC in which the identified chemical is controlled.
      a) Substances controlled by structural class where data generated cannot distinguish between isomers that are controlled under the same section, isomer determination is not required for reporting.
      b) If the isomer is determined, it may be reported.
   3. For example: Contains fluoro-ABICA: A compound with an Indole (Core), Amino oxobutane (Group A) and Carboxamide (Link)
      Note: This substance is controlled in accordance with Texas HSC 481.1031, subsection (b)(5).

D. Reporting Cactus-like Samples
   1. Plants visually consistent with Peyote (Lophophora spp) will be analyzed for the presence of mescaline.
   2. If mescaline is identified it will be reported as “Contains Mescaline,” with the following result note:
      “The Laboratory is unable to determine if plant material is peyote.”
E. Reporting Opium Samples

1. Morphine, codeine, and thebaine are the opium alkaloids that are controlled substances. Non-controlled alkaloids include papaverine, noscapine and narceine. Opium samples, including commercial preparations such as Paregoric, should be reported as “Contains Opium” only if there is no heroin present and morphine and codeine are detected in combination with at least one of the other alkaloids.

2. Alternatively, the results can be reported as “Contains Codeine and Morphine and (at least one other major alkaloid)” with a footnote stating: “These are commonly detected constituents of opium.”

2.2 Reporting No Analysis, No Controlled Substances Detected, Dangerous Drugs, Non-Controlled Substances, Inconclusive, and Preliminary Results.

A. No Analysis Results

1. Exhibits that are not examined chemically, microscopically, or subjected to pharmaceutical identification are reported as “No Analysis” or “No Analysis Requested”.

2. An appropriate footnote may be chosen at the discretion of the analyst.

B. No Controlled Substances Detected Results

1. If a sample is subjected to sufficient testing and a controlled substance is not detected, the sample will be reported as “No controlled substance detected” unless the analyst chooses to report other substances that are confirmed.

   Sufficient testing will be met minimally by testing for basic and acidic drug, and shall include at least two tests, of which one must be confirmatory.

2. An appropriate footnote may be chosen at the discretion of the analyst.

3. If after sufficient testing, peaks are present to indicate the presence of a compound(s), but neither a controlled substance nor a non-controlled substance is suspected, the sample will be reported as “No controlled substance detected.”

4. If after sufficient testing, a controlled substance is suspected but unable to be confirmed, refer to “Inconclusive Results” for reporting options.

C. Dangerous Drugs Results

1. If after testing, a dangerous drug is identified, it should be reported.

2. An appropriate footnote may be chosen at the discretion of the analyst.

3. If after testing, a dangerous drug is suspected but unable to be confirmed, refer to “Inconclusive Results” for reporting options.

D. Non-Controlled Substances Results

1. If after testing, a non-controlled substance is identified, it may be reported.

2. An appropriate footnote may be chosen at the discretion of the analyst.

3. If after testing, a non-controlled substance is suspected but not confirmed, refer to “No Controlled Substances Detected Results” for reporting options.
E. Inconclusive Results

1. If testing indicates the presence of a substance that cannot be identified, the results may be reported as “Unable to identify”, with the appropriate accompanying footnote.
   a) For insufficient amount of evidence: “Insufficient sample for identification”
   b) For insufficient instrumentation: “Due to limitations in instrumentation, the laboratory is unable to identify the compound in this exhibit at this time.”
   c) For no reference standard or reference library available: “Due to unavailability of appropriate reference standard/reference libraries, the laboratory is unable to identify the compound in this exhibit at this time.”
   d) For unsuitable evidence (e.g., decomposed plant material) for identification: “Due to the unsuitable condition of the evidence, the laboratory is unable to identify the substance.”

F. Preliminary Results

1. For pharmaceutical exhibits other than alprazolam, hydrocodone, or oxycodone: If only visual pharmaceutical examinations are performed and confirmatory testing has not been performed, the results will be reported as follows:
   a) “Only a preliminary pharmaceutical observation was performed. In reference to published data, the item is visually consistent with information that it contains [drug name]. This observation was not confirmed by instrumental analysis.” (Controlled Substances or Dangerous Drugs)
   b) “Only a preliminary pharmaceutical observation was performed. In reference to published data, the item is visually consistent with information that it contains no controlled substance or dangerous drug. This observation was not confirmed by instrumental analysis.”
   c) “Only a preliminary pharmaceutical observation was performed. No published data was found. No further tests are being performed at this time.” Note: this statement can only be used when it is not the only item in the case.

2. For alprazolam, hydrocodone, or oxycodone and non-pharmaceutical exhibits: If preliminary examinations are performed other than weight, the analytical process must be completed.

2.3 Reporting Chemicals Associated with Manufacture of Controlled Substances

A. For chemicals listed in HSC 481.124 associated with manufacture (examples include but are not limited to anhydrous ammonia, water samples containing ammonia, red phosphorus, lithium, and iodine):

1. If the substance has been confirmed, the substance will be reported as “Contains [substance]”.

2. If the substance has not been confirmed, the substance will be reported as “Contains a chemical associated with manufacture” followed by an appropriate footnote, such as: “The appearance of the sample and the results of presumptive test(s) indicate that this exhibit contains [substance].”

B. No weight is required for these types of samples, although one may be reported if desired.
2.4 Reporting Weights and Uncertainty

A. The reported conclusion must be applied to either the net weight or gross weight.

B. A net weight must be reported for each exhibit, except under the following circumstances: (At the analysts’ discretion, a net weight may be reported for the following circumstances.)
   1. Exhibits which will not be analyzed
   2. Samples from a larger exhibit submitted to the laboratory for analysis. [Customer collected samples from bulk evidence]
   3. Exhibits containing only compounds listed in penalty group 1-A when abuse units can be counted
   4. Exhibits which are reported using gross weight
   5. Exhibits which are re-examined as part of a quality assurance process.

C. Top loader balance weights will be reported in grams. Bulk scale weights will be reported in kilograms or pounds, as weighed.

D. All reported weights will be truncated to two decimal places (except as specified in H.1 below) and will include the appropriate units of measurement with the expanded measurement uncertainty (except as specified in F below).

E. The expanded uncertainty will be rounded up to the same number of decimal places as the reported weight.

F. Circumstances when the uncertainty may not be reported:
   1. Reported weight is less than or equal to the expanded uncertainty
   2. Reported sampled weight for a statistical sampling plan
   3. Reported sampled weight for a pharmaceutical sampling procedure
   4. Reported submission weight
   5. Customer requested additional weights

G. Report the weight of substances identified in grams, except for the following:
   1. Weights greater than or equal to 1,000 grams may be reported in kilograms, when appropriate (e.g. weighed on a bulk scale).
   2. Weights less than 0.01 grams must be reported as “Trace”.

H. Report the net weight of plant material identified as Marihuana or synthetic cannabinoids:
   1. Weights greater than one pound should be reported in pounds to at least one decimal place.
   2. Weights less than 0.01 grams must be reported as “Trace”.
   3. If a marihuana sample weighs less than 0.01 ounces, the analyst may report the weight in grams with a footnote stating, “Less than 0.01 ounces”.
   4. To convert grams to ounces, use 28.4 grams per one ounce.

I. Report the weight of liquid samples if a controlled substance or dangerous drug is identified. The volume may be reported; however, an uncertainty for volume will not be calculated.
J. For compounds which are in an inseparable carrier matrix (e.g., PCP laced cigarettes), the entire weight of the exhibit will be reported with a footnote stating “The net weight includes the carrier matrix”.

K. The gross weight of substances identified may be reported only after tare weight has been evaluated using SD-03-01.

L. Submission weight may be reported, upon request.

2.5 Reporting Quantitation Results of Methamphetamine and Uncertainty

A. The quantitation result is reported with the net weight of the exhibit.

B. If quantitation results are calculated above 100%, report the result as is, provided the results are within uncertainty.
   1. If the results are outside of accepted uncertainty, the quantitation must be repeated.

C. All quantitated items will include results in salt form with the appropriate units, expanded measurement uncertainty calculation with appropriate matching units, and the coverage percentage of the uncertainty.

D. The quantitation reported value shall be the average of the results from the two samples.
   1. In order for the LIMS to accurately report the expanded measurement uncertainty calculation with the correct number of decimal places:
      a) Truncate the quantitation value to one decimal place when the expanded measurement uncertainty calculation is equal to or greater than 1 and less than 10.
      b) Truncate the quantitation value to two decimal places when the expanded measurement uncertainty calculation is less than 1.

E. The expanded measurement uncertainty is rounded up to the same number of decimal places as the reported result.

F. Only solid samples will be quantitated and the quantitation results will be reported as a percentage.
   1. The results of the two independent samples must be within 5% of each other in order to report.
   2. Percent difference is calculated by:

\[
Percent\ Diff = \frac{|x_1 - x_2|}{\frac{x_1 + x_2}{2}} \times 100
\]

G. The purity of the hydrochloride salt is determined.
   1. The conversion calculation is determined using the ratio of the molecular weight of the salt form to the base form.
   2. Clarke’s Isolation and Identification of Drugs or Clarke’s Analysis of Drugs and Poisons will be utilized to obtain molecular weight data.
   3. Example for Methamphetamine:

\[
\frac{185.7\ \text{methamphetamine HCl}}{149.2\ \text{methamphetamine}} = 1.24
\]

   \[
   \text{Percent Methamphetamine HCl} = 1.24 \times \text{identified base purity}
   \]
2.6 Reporting the Number of Abuse Units for Substances in Penalty Group 1-A

A. Report abuse units as defined in HSC 481.002(50).
   1. Count and report the number of perforated blotter paper, tablets, gelatin wafers, sugar cubes, stamps, or other single abuse units.
   2. If the blotter paper is not marked, each one quarter-inch square section of paper is considered a single abuse unit.
   3. If the sample is liquid or solid and not divided into units, 40 micrograms is considered to be one abuse unit.

B. When abuse units are determined using a calculation, report the net weight with its associated uncertainty.

2.7 Reporting the Use of a Statistical Sampling Plan

If a statistical sampling plan is used, the confidence level and inference to population must be included on the report.

2.8 Reporting of Pharmaceuticals Using a Non-Statistical Sampling Procedure

When a statistical sampling plan is not used, a description of the number of items sampled for analysis, results, and the weight of items sampled must be included on the report.
SD-01-02 MEASUREMENT UNCERTAINTY ESTIMATE – WEIGHTS

1 Scope
Measurement uncertainty for the reporting of weights of seized drug evidence based on the type of balance or scale used.

2 Related Chapters
Reporting Guidelines (SD-01-01)
Examination of Seized Drug Evidence (SD-03-01)

3 Standards, Controls, and Calibration
Calibration of a balance or scale is performed by an approved vendor annually.

4 Practice
A. A system-wide measurement uncertainty estimate, which conveys approximately 95% confidence (coverage factor k=2), is determined using data submitted from all DPS laboratories to create a combined measurement uncertainty budget.
B. The measurement uncertainty estimate is included in the case record and/or LIMS.
C. The uncertainty estimate is evaluated on an annual basis and updated when needed.
D. The measurement uncertainty is based on:
   1. System reproducibility
   2. Balance linearity
   3. Balance uncertainty

Current Estimates

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<td>Top Loading</td>
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<tr>
<td>Bulk</td>
<td>0.063 lbs</td>
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5 Calculation
A. Static and Dynamic Weighing
   1. Using a single balance (LIMS performs this calculation)
      \[ U = \sqrt{\# Weighing Events} \times Expl\ expanded\ Uncertainty(Balance\ Type) \]
   2. Using multiple balances (hand calculation required, unit conversion may be necessary, LIMS does not perform this calculation)
      \[ U = \sqrt{\# Weighing Events}_{(Balance\ Type\ 1)} \times Expl\ expanded\ Uncertainty_{(Balance\ Type\ 1)} + \sqrt{\# Weighing Events}_{(Balance\ Type\ 2)} \times Expl\ expanded\ Uncertainty_{(Balance\ Type\ 2)} \]
B. Complex Weighing (single balance only; LIMS performs this calculation)

\[
U = 2 \sqrt{\left[ (\sqrt{\#\text{Weighing Events}} - 1) \times \sigma_{\text{Balance Type}} \right]^2 + \left( \#\text{Packages} \times \sigma_{\text{Balance Type}} \right)^2}
\]

6 Literature References and Supporting Documentation

Annual System Seized Drugs Uncertainty Budget Validation
SD-01-03  MEASUREMENT UNCERTAINTY ESTIMATE – QUANTITATION

1 Scope
Measurement uncertainty for the reporting of quantitation of controlled substances based on the procedure for Gas Chromatography and Liquid Chromatography.

2 Related Chapters
Reporting Guidelines (SD-01-01)
Examination of Seized Drug Evidence (SD-03-01)
Quantitation by Gas Chromatography with Internal Standard (SD-07-01)
Quantitation by High Performance Liquid Chromatography (SD-07-02)
Quantitation Sampling Procedure (SD-07-03)

3 Practice
A. A system-wide measurement uncertainty estimate, which conveys approximately 95% confidence (coverage factor k=2), is determined using data across from DPS laboratories conducting quantitation to create a combined measurement uncertainty budget.
B. The measurement uncertainty estimate is included in the case record and/or LIMS, such that it can be added to the reported quantitation value.
C. The uncertainty estimate is evaluated on an annual basis and updated as needed.
D. The measurement uncertainty is based on:
   1. System reproducibility
   2. Sample homogeneity
   3. Purity of the Reference Material (Calibrator)

4 Calculation
A. Combined Standard Uncertainty =

\[
\sqrt{\left(\text{std unc reproducibility data}\right)^2 + \left(\text{std unc sample homogeneity}\right)^2 + \left(\text{std unc purity calibrator}\right)^2}
\]

\(\text{std unc}\) = standard uncertainty value obtained from current validation data
B. The expanded measurement uncertainty at k=2 is the combined standard uncertainty multiplied by 2.

5 Literature References and Supporting Documentation
System Uncertainty Budget Validation Summaries
SD-01-04  CASE DOCUMENTATION

1  Scope
These policies are established as minimum requirements for additional case documentation and record-keeping required for seized drugs cases.

2  Practices
2.1  Case Record
   A.  Evidence Record Sheet (optional)
   B.  Laboratory Submission Form (LAB-201)
   C.  Analyst Notes
       1.  Analyst notes containing additional information about the evidence, analysis procedures or any other explanatory notes may be recorded on separate sheets.
       2.  Each page will include the case number, handwritten initials or secure electronic equivalent, and the date the notes were recorded.
       3.  Page numbers may be added.
   D.  Analytical Data
       1.  Electronic instrumental data shall not be deleted and should be backed up periodically to avoid potential loss.
       2.  All charts, spectra, notes, and photographs will be maintained and archived in accordance with the Laboratory Records chapter of the CLS Manual.
       3.  If solvent blanks were run on GC/MS prior to a trace sample, then the charts will be maintained in the case record.

2.2  Exam Counting Guidelines
   A.  Examinations for seized drug casework must be counted and recorded on the Seized Drugs Worksheet (LAB-SD-01) or in LIMS under the Examinations tab. Total exam count does not need to be calculated or entered into LIMS.
   B.  Number of Items is the actual number of individual items received (e.g., 10 tablets = 10; two baggies of marihuana = 2; 1 sheet of 25 squares of LSD = 1; plastic corner baggie of 10 crack rocks = 1 item).
   C.  Number of Items Tested is the actual number of individual items on which both preliminary and confirmatory examinations were performed.
   D.  Pharmaceutical identification of 500 tablets is considered one (1) for Analysis and one (1) for Number of Items Tested, not 500.
   E.  The examination of a sheet of 250 LSD squares is equal to one (1) for Number of Items Tested and is equivalent to one (1) for Analysis, not 250.
SD-01-05 STANDARD ABBREVIATIONS LIST

1 Scope
This document lists abbreviations commonly used in the Seized Drugs discipline. Abbreviations may appear as uppercase or lowercase in case records and may be made plural with the addition of “s” or “’s”.

2 Abbreviations

2.1 Color
- B blue
- BLK black
- BRN brown
- GRN green
- O orange
- P purple
- PK pink
- R red
- V violet
- WHT white
- Y yellow

2.2 Other
- acid 0.2 N sulfuric acid
- base 1 M sodium carbonate
- comp composite, composition, or combination
  → contains, containing, placed inside, into
- cryst crystal(s); crystalline
- dk dark
- env envelope
- evid evidence
- ex exhibit
- inc inconclusive
- insuf insufficient
- ISTD internal standard
- Lt light
- NA not analyzed, no analysis
- NCS no controlled substance detected
- NSM non-statistical method
- PDMABA para-dimethylaminobenzaldehyde
- pharm pharmaceutical identification
- PS plant substance
- pl plastic
<table>
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<tr>
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<th>Meaning</th>
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<tr>
<td>Rep</td>
<td>representative or representative sample</td>
</tr>
<tr>
<td>RT</td>
<td>retention time</td>
</tr>
<tr>
<td>Rx</td>
<td>prescription</td>
</tr>
<tr>
<td>SSP</td>
<td>statistical sampling plan</td>
</tr>
<tr>
<td>std</td>
<td>standard</td>
</tr>
<tr>
<td>subst</td>
<td>substance</td>
</tr>
<tr>
<td>unk</td>
<td>unknown</td>
</tr>
<tr>
<td>wk</td>
<td>weak</td>
</tr>
<tr>
<td>&lt;&gt;</td>
<td>opposite side</td>
</tr>
</tbody>
</table>
SD-01-06 SEIZED DRUGS OVERVIEW

1 Scope
To establish general policies for the analysis of seized drug evidence.

2 Practices

2.1 Routine Examination
Routine drug identification may include any or all of the following at the discretion of the laboratory:

A. Determination of net or gross weight, chemical screening examinations, instrumental confirmation tests, and pharmaceutical identification of tablets/capsules.

B. Some exhibits may not be analyzed depending on circumstances of the case.

C. Photograph and/or repackage evidence.

D. DPS evidence submissions of excess quantity cases will follow laboratory policies for destruction.

E. Evidence previously analyzed will not typically be re-analyzed except by court order or under the following circumstances:
   1. Administrative or quality assurance purposes
   2. Original forensic scientist is not available for testimony
   3. New technology or procedures become available

F. The Laboratory may consider a resubmission of evidence for analysis using a different test and/or processing of additional evidence.

G. The reason for reanalysis will be documented in the case record.

H. Syringes typically are not examined without a request from the prosecutor's office.

I. Laboratories do not provide services for destruction of hazardous materials.

2.2 Examiner Approval and Assessment

A. Demonstration of competency in the use of seized drugs procedures is required prior to work.

B. Authorization in the Seized Drugs discipline is granted for the following categories of test method(s) or relevant testing procedure:

   For Qualitative and/or Quantitative
   1. Chemical Testing
   2. Physical Examination
   3. Instrumental Analysis
02 QUALITY ASSURANCE FOR DRUG ANALYSIS
SD-02-01 STANDARDS AND REFERENCES

1 Scope
These policies serve to establish guidelines for the use of drug reference standards and libraries.

2 Practices

2.1 Drug Reference Standards

A. Drug reference standards will be recorded in a permanent logbook detailing a complete inventory of each drug in stock. The log will contain
   1. name of the drug,
   2. date received or used,
   3. initials or name of the person making the entry in the log,
   4. source from which the drug was obtained,
   5. purpose for which it was used,
   6. lot or identification number if available,
   7. form or concentration,
   8. quantity,
   9. balance remaining, and
   10. disposal date.

B. The quantity of drug reference standards to be stored in each laboratory may be determined by the Quality Manager, based on the need and request of each laboratory.

C. Drug reference standards will be stored in a securely locked container with only persons authorized by the appropriate supervisor having access.

D. Drug reference standards will be inventoried and documented at least annually.
   1. All reference standards must be accounted for and any substance in Texas Schedule I or II must have its quantity verified against the logbook.
   2. Any significant quantity differences will be evaluated by the TPOC, Supervisor, or Quality Manager and the evaluation will be documented.
   3. Corrective action through the QI/QAP process may be necessary following the evaluation.

E. These provisions are not intended to prevent analysts from having access to small quantities of drug standards at the bench for routine use in analyses.

F. Internal reference standards are thoroughly analyzed and characterized before use as standards or controls.

2.2 Quality Control Procedures for Drug Standards

A. Before using a new drug standard, an FTIR or GC/MS analysis will be performed in order to verify that the compound is what it is purported to be.

B. The resulting spectra will be placed in a quality control book and labeled with all pertinent information, such as the lot number, source, and initials of the chemist who performed the analysis.
C. Some commercially prepared drug standards are mailed with GC/MS analysis data and other quality control data. These data sheets will be retained.

### 2.3 Verification of Laboratory Generated Standards

A. Thoroughly analyze and characterize any in-house samples before they are used as a standard or reference.

B. The identity of the substance must be confirmed by FTIR and/or GC/MS and verification data retained by the laboratory before it can be used as a reference, if a compound:
   1. Must be synthesized by a chemist in the laboratory, or
   2. Is obtained from another source (e.g. another lab).

C. The Quality Manager will determine when adequate verification has been completed on any compound to be used as a reference sample.

### 2.4 Source References

A. When analyzing compounds using either GC/MS or FTIR, the spectra will be compared to a reference standard. The source of the spectrum of this standard will be documented in the case record.

B. References used for pharmaceutical identification will be documented in case record. A list of commonly used pharmaceutical references and their approved abbreviations are found in SD-02-03.

C. The Approved List of Reference Libraries and Abbreviations (SD-02-02) will be used to denote the common references for standard spectra. Addition of other routine references may be added with the approval of the Seized Drugs Advisory Board.

### 2.5 Reference Spectral Libraries

A. Reference libraries of spectra used in identification of compounds must be fully documented, uniquely identified, and properly controlled.

B. Commercial libraries of mass spectra and infrared spectra in electronic form that were acquired from external sources for use with the laboratory’s analytical instrumentation meet these requirements, as do published reference collections and reputable scientific literature.

C. For reference libraries produced by the laboratory, at least one of the following requirements must be met for each entry used to confirm the identity of seized drug evidence:
   1. The compound used to generate the spectrum must be traceable to the lot number of the appropriate standard. The analyst that generates the spectrum must note, either on the reference spectrum itself or with the information that accompanies it, the manufacturer’s or supplier’s company name and lot number, the date the entry was generated, and his/her initials; or
   2. The spectral data in the entry must be matched to data for the same compound that is published in an approved library or literature. The analyst that performs the comparison must note, either on the reference spectrum itself or with the information that accompanies it, the date the match was verified, the source of the reference used for the comparison, and his or her initials.
### SD-02-02  APPROVED LIST OF REFERENCE LIBRARIES AND ABBREVIATIONS

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Resource</th>
</tr>
</thead>
<tbody>
<tr>
<td>CND</td>
<td>CND Analytical</td>
</tr>
<tr>
<td>G</td>
<td>Georgia Bureau of Investigation (Georgia State Crime Lab) Library</td>
</tr>
<tr>
<td>J</td>
<td>Journal (Microgram, CLIC, Journal of Forensic Science, etc.)*</td>
</tr>
<tr>
<td>L</td>
<td>In-house Laboratory library</td>
</tr>
<tr>
<td>M</td>
<td>Instrumental Data for Drug Analysis (Mills, et al.)</td>
</tr>
<tr>
<td>N</td>
<td>NIST (NBS) Library</td>
</tr>
<tr>
<td>A</td>
<td>American Academy of Forensic Science Drug Library</td>
</tr>
<tr>
<td>C</td>
<td>Clarke’s Isolation and Identification of Drugs</td>
</tr>
<tr>
<td>CP</td>
<td>Clarke’s Analysis of Drugs and Poisons</td>
</tr>
<tr>
<td>IC</td>
<td>Mattson ICON Library</td>
</tr>
<tr>
<td>PMW</td>
<td>Pfleger/Mauer/Weber Drug Library</td>
</tr>
<tr>
<td>AL</td>
<td>Aldrich Library of IR Spectra</td>
</tr>
<tr>
<td>FL</td>
<td>Fluka IR Library</td>
</tr>
<tr>
<td>GG</td>
<td>Goldgate ATR library</td>
</tr>
<tr>
<td>DPS</td>
<td>DPS Chemical Library</td>
</tr>
<tr>
<td>SWGM</td>
<td>SWGDRUG Monographs</td>
</tr>
<tr>
<td>SWG</td>
<td>SWGDRUG MS Library Version 3.2 or later</td>
</tr>
<tr>
<td>FX</td>
<td>Forendex** (can be found online)</td>
</tr>
<tr>
<td>CSL</td>
<td>Cayman Spectral Library** (can be found online)</td>
</tr>
<tr>
<td>DD</td>
<td>Designer Drugs Online Mass Spectral Database** (can be found online)</td>
</tr>
<tr>
<td>EPR</td>
<td>European Project Response** (can be found online)</td>
</tr>
</tbody>
</table>

**Note:** If the reference used has several volumes/versions, then the analyst may choose to indicate the particular volume/version as part of the abbreviation. For example, NIST 12 and NIST 62 may be indicated as N12 and N62 respectively.

* Use a footnote in the case record to indicate which journal was used and the literature citation.

** For standard verification purposes only.
### SD-02-03 APPROVED ABBREVIATION LIST FOR PHARMACEUTICAL REFERENCES

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Resource</th>
</tr>
</thead>
<tbody>
<tr>
<td>PDR</td>
<td>Physician’s Desk Reference</td>
</tr>
<tr>
<td>LOGO</td>
<td>DEA Logo Index</td>
</tr>
<tr>
<td>ID</td>
<td>Ident-a-drug</td>
</tr>
<tr>
<td>RX</td>
<td>Rx List, http:\www.rxlist.com</td>
</tr>
<tr>
<td>RXID</td>
<td>Amer-Chem Rx-ID</td>
</tr>
<tr>
<td>DEF</td>
<td>Diccionario de Especialidades Farmacéuticas</td>
</tr>
<tr>
<td>PC</td>
<td>Poison Control</td>
</tr>
<tr>
<td>DIB</td>
<td>Drug ID Bible</td>
</tr>
<tr>
<td>DC</td>
<td>Drugs.com, http:\www.drugs.com</td>
</tr>
<tr>
<td>Pharm</td>
<td>Pharmaceutical Identification from packaging or manufacturer information</td>
</tr>
</tbody>
</table>
SD-02-04 REAGENTS

1 Scope
To establish quality assurance guidelines for reagents, chemical preparations and solvents used in seized drug analysis.

2 Safety
A. Use caution when handling any unknown substance or chemical.
B. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
C. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Practices
A. All reagents may be prepared in any desired volume as long as the proportions specified in the individual instructions are maintained; however, the volume must be sufficient to allow for necessary performance testing.
B. All prepared reagents will be labeled with the identity of the reagent, the preparer’s initials, and the date of preparation or lot number. Records shall be maintained identifying who made the reagent and the components used in preparation.
C. All reagents will have an expiration date of two years from the date of preparation, unless otherwise defined in the respective reagent's preparation instructions.
D. For prepared reagents (including simple dilutions) that are not performance checked, a reagent preparation logbook will be maintained that includes at least the following information:
   1. Name of the solution
   2. Preparation date
   3. Initials of the individual who prepared the reagent
E. For each reagent that requires a performance check prior to use in casework, a reagent quality control logbook or equivalent notation in the case record will be maintained that includes at least the following information:
   1. Name of the solution
   2. Preparation date
   3. Initials of the individual who prepared the reagent
   4. Date the reagent was quality tested
   5. Performance result, documented in the same manner as the case record.
   6. Initials of the person who tested the reagent
F. Reagents which are subject to a performance check and are stored will have a performance check completed every month, unless:
   1. The individual reagent’s instructions specify a shorter interval or
   2. The reagent has not been used for a month or more
G. Standards and Controls
   1. Reagents will be checked using appropriate standards and controls.
   2. If the reagent does not perform as expected, the reagent will be discarded and a fresh reagent will be prepared and performance checked using a known standard.
   3. No reagent or other chemical preparation will be used in casework if it is not working properly or if it is contaminated.

H. The following reagents and/or reagent systems are subject to a performance check:
   1. Color Test Reagents
      a) Cobalt Nitrate
      b) Scott/Cobalt Thiocyanate
      c) Chlorophenol Red: Modified Schweppe’s
      d) p-Dimethylaminobenzaldehyde
      e) Duquenois-Levine
      f) Ferric Chloride
      g) Formaldehyde-Sulfuric Acid
      h) Janovsky
      i) Liebermann
      j) Marquis
      k) Modified SNP
      l) Weber

   2. TLC Indicator Reagents (verified when performing Thin-Layer Chromatography, CS-06-01)
      a) Acidified Iodoplatinate
      b) Fast Blue RR
      c) Ninhydrin
      d) Potassium Permanganate
      e) p-DMABA
      f) Marquis

4 Records
Logs or equivalent documentation
SD-02-05 QUARTERLY EVIDENCE RE-EXAMINATION

1 Scope
The seized drugs evidence reexamination is a quality assurance process to monitor laboratory performance by comparison of results internally.

2 Related Documents
CLS Manual: Monitoring the Validity of Results

3 Safety
A. Use caution when handling any unknown substance or chemical.
B. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
C. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.
D. Use proper lifting techniques and caution when handling heavy items.
E. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

4 Practices
A. At least one released seized drugs case per analyst will be selected for re-examination, according to the calendar year quarter in which the case report was released.
B. Every analyst should work at least one single-item DPS case each quarter. This ensures that cases are available for reexamination and are not unduly complex.
C. The evidence will be re-examined using appropriate procedures and the analytical process will be properly documented. The re-examining analyst may choose to follow the same analysis procedures as the original analyst.
D. The cases selected each quarter for re-examination must be completed and assessed by the end of the following quarter. (Example: Cases subject to reexamination in quarter 1 must be re-examined and assessed by the end of quarter 2.)
E. The laboratory must compile a list of cases that were re-examined each quarter, including the following information for each case:
   1. Case number
   2. Name of the original analyst,
   3. Date the original report was issued,
   4. Name of the reexamining analyst,
   5. Date the reexamination was completed, and
   6. Assessment of the reexamination results.
F. If an analyst does not perform any analysis in a particular quarter, that fact should also be documented in the list.
5 Assessment Criteria

A. The substance identified on re-examination must agree with the substance identified in the original analysis.

B. The net weight shall be documented in the case record, but not on the report of re-analyzed exhibits, and should be within 10% of the original after-analysis weight (or within 10% of the net weight, if no after-analysis weight was documented).

C. Inconsistencies >10% shall be evaluated and documented in the case record. If necessary, a Quality Incident/Action Plan may be initiated.

6 Reexamination report

A. A supplemental report will be issued to the original requesting authority:

B. If the conclusions of the evidence re-examination are consistent with the originally reported conclusions, a report will be issued with a result stating, "This exhibit was re-examined for quality assurance purposes. The results are consistent with the original conclusions."

1. If the original analyst is not available, an additional supplemental report with weights and conclusions may be issued by the re-examining analyst.

C. If the conclusions are not consistent with the original report:

1. The Laboratory/Quality Manager, or designee, will initiate a Quality Incident to investigate the issue further.

2. If it is determined that the original analysis is incorrect:

   a) A supplemental report will be issued stating, "This exhibit was re-examined for quality assurance purposes. The results of re-examination are inconsistent with the original report. An amended report correcting the discrepancy will be issued."

   b) An amended report will be issued by the original examiner correcting the inconsistency.

   c) If the original analyst is not available, the amended report will be issued by the re-examining analyst.

3. If it is determined that the re-examination analysis is incorrect, the inconsistency will be corrected and a supplemental report will be issued stating, "This exhibit was re-examined for quality assurance purposes. The results are consistent with the original conclusions."

7 Records

A. Evidence Re-examination List

B. Re-examination records in respective case record

C. Records of the annual inspection will be maintained in the laboratory and copies will be provided to the Laboratory Director.
SD-02-06  GUIDELINES FOR TECHNICAL REVIEW

1  Scope
The technical review is a documented review of the entire case record prior to the release of results. The technical review is conducted by an individual currently or formerly qualified to perform technical review in the Seized Drugs discipline, as outlined in the Crime Laboratory Service Manual.

2  Related Documents
CLS Manual: Review of Laboratory Records

3  Practices

3.1  Items for Review
A. Case Record
Ensure that all required documents are present and documents generated by the laboratory are labeled appropriately with the case number, exhibit number (if applicable), handwritten or electronic initials, and the date generated.

B. Worksheet
1. Ensure that the description of the evidence matches the information entered on the submission form. Verify the documentation of any observed discrepancies.
2. Verify that an appropriate balance was selected.
3. Verify all calculations performed.
4. Ensure all required weights have been documented and are appropriate.
   a) If optional weights are documented, ensure that they are appropriate.
   b) For reported weights or quarterly re-examinations, ensure that the number of weighing events has been recorded.
5. Verify that an appropriate sampling plan was selected and properly applied.
6. If performed, ensure that color test results have been appropriately documented.
7. If performed, ensure that the pharmaceutical identification was made with an approved reference.
8. If performed, ensure that the documentation for both macroscopic and/or microscopic exams are appropriate and support the reported result.
9. If performed, ensure that the results for TLC have been appropriately documented.
10. Ensure that all analysis notes are accurate and appropriate.
11. Verify that any composite samples used are documented appropriately.

C. Data
1. Verify that if an extraction has been performed, it has been documented and is appropriate.
2. Ensure that the required instrumental blanks have been performed and that they do not contain a dangerous drug or controlled substance.
3. Verify that the result obtained has been evaluated and matched to an approved reference.
4. If applicable, ensure that the retention time of the sample is within 1% of the standard.
5. Verify that all peaks evaluated are appropriate and labeled correctly.
6. Verify that the method(s) used is/are appropriate for the compound(s) tested.

D. Results
1. Verify that the correct result has been selected from the list.
2. Ensure that sufficient testing has been performed to support the reported result.
3. Ensure that the reported results are supported by the data obtained.

E. Report
1. Verify that the correct number of items and number of items tested have been added to the appropriate exhibits.
2. Verify the uncertainty is reported, as required.
3. If applicable, verify that the appropriate result footnote(s) have been added.
4. If applicable, verify that the report type (amended/supplemental) is appropriate.

F. Other
1. Excess Quantity Cases
   a) Verify that excess quantity cases are sampled correctly, labeled properly, the exemplar has been separated out from bulk and recorded, and at least one photograph (treated as evidence) has been included.
   b) Verify that the appropriate net weight and gross inventory weight are documented per SD-03-04.
2. Quantitation Cases
   Ensure that all procedures have been followed for quantitation cases per the SOP.

4 Records
The technical review will be documented in the LIMS.
03 EXAMINATION OF SEIZED DRUGS

SD-03-01 EXAMINATION OF SEIZED DRUG EVIDENCE

1 Scope
To describe a basic analytical scheme to weigh, utilize screening tests, extraction techniques, and instrumental analytical procedures for the isolation and identification of seized drug evidence.

2 Related Chapters
Standard Abbreviations List (SD-01-05)
Instructions for Seized Drugs Worksheet (SD-03-02)
Critical Weights (SD-03-03)
Examination and Destruction of Excess Quantity Seized Drug Property (SD-03-04)
Physical Examination of Canabis sativa L. (SD-03-05)

3 Safety
A. Use caution when handling any unknown substance or chemical.
B. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
C. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.
D. Use proper lifting techniques and caution when handling heavy items.
E. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

4 Procedure
Document the observations and examinations in the Laboratory Information Management System (LIMS), or on the approved seized drugs worksheet (LAB-SD-01) if LIMS is unavailable. Other supplemental documents may be used.

4.1 Retrieval and Initial Examination of the Evidence
When an item of evidence requires analysis by multiple disciplines, the forensic scientist is responsible for ensuring the evidence is processed in the proper order before examinations begin.

1. Retrieve the evidence following laboratory policy concerning barcodes and the transfer of the items to the analyst.
2. Visually examine the evidence and inventory the items. Compare the information on the Laboratory Submission Form to the physical evidence and ensure all information is consistent regarding the description of the evidence, condition of seals, and case number.

   a) If an item contained within tamper-resistant packaging cannot be visualized through the innermost packaging and is not going to be analyzed, it is acceptable to omit the inventory of the item. The outer package of this item will still need to be documented in LIMS.
   b) Any identified discrepancies must be documented.
c) If the noted differences may impact the requested service, then the submitting official shall be notified of the inconsistency before work commences. The communication must be documented.

d) If wet plant material is submitted, it should be air dried in a secure location of the laboratory, then re-packaged into appropriate containers such as paper bags.

3. Separate (sub-divide) exhibits with multiple items, including tablets, if the contents appear to be different (e.g. color, imprint, shape, and size).
   a) Care must be exercised to avoid cross-contamination if more than one item of evidence is open at the same time.
   b) Sub divide exhibits based on visual differences unless:
      i. The items within the exhibits are not to be analyzed.
      ii. All items within the exhibit are confirmed individually.
      iii. The exhibits include edibles in factory packaging (sealed or unsealed) and there is a reasonable expectation of homogeneity. In this event, the items do not have to be sub-divided based upon the color, imprint, or shape.

4. Enter a brief description of each item and mark the examined items with case number and analyst’s initials.
   a) The marking of a proximal layer of packaging containing multiple items is acceptable as long as all the items belong to the same exhibit.
   b) The use of abbreviations is acceptable as long as they are widely used in the same manner as defined for the relevant scientific community, from an authorized list (e.g. SD-01-05, Standard Abbreviations List), or have been defined in the case notes.
   c) Large numbers of tablets or capsules may be approximated using weight calculations.
      i. Logos and/or significant markings may be documented and compared with pharmaceutical reference literature.
      ii. Document the reference used for the comparison.
      iii. Information found on untampered pharmaceutical packaging may be used as pharmaceutical identification.

5. Repackage items as necessary. Document any repackaging done by the analyst or significant changes to the evidence.

4.2 Procedure for Weighing Samples

A. General
   1. Select the appropriate balance for the amount of sample to be weighed.
   2. Indicate the specific balance used in LIMS.

B. Submission Weight
   1. A submission weight is defined as the weight of separately submitted containers and their contents.
   2. A submission weight is taken prior to testing or examination.
C. Before Analysis Weight
   1. May include or exclude packaging; document appropriately.
   2. If the before analysis weight includes weight of packaging, a tare weight must be obtained in order to calculate a net weight, unless appropriate footnote is used.

D. Tare Weight
   1. Tare weight is the weight of an empty container.
   2. Determining a tare weight
      a) A tare weight is defined as the total weight of the packaging that was included in the before analysis weight.
      b) When multiple items are grouped in multiple weighing events for direct tare weight determination, the items in each group must be documented.
         Example: 6 bags with two weighing events may be documented as:
         \[2.40 \text{ g (bags 1-2)} + 2.57 \text{ g (bags 3-6)} = 4.97 \text{ g tare weight}\]
      c) If the calculated net weight, including the uncertainty, does not encompass a critical weight limit defined in Critical Weights (SD-03-03), estimation of tare weight is allowed.
         i. When all the packaging is virtually identical (size, thickness, and sticker labels), one can be weighed and multiplied by the total number of packages.
         ii. When all the packaging is not virtually identical, the item may be sub-itemized into virtually identical groupings and processed as above.
      d) If the calculated net weight, including the uncertainty, encompasses a critical weight limit defined in Critical Weights (SD-03-03), then 100 percent tare weight must be performed.
   3. Documenting a tare weight
      a) The tare weight must be in same units as the before analysis weight.
      b) Conversion factor used is 28.4 g/oz or 454.4 g/lb.
      c) Do not truncate values.
      d) If the source of tare weight is not obvious, document how the tare weight was calculated or estimated.

E. Net Weight
   1. Net weight is the weight of an item without the weight of its packaging. It is calculated by subtracting the tare weight from the gross or before analysis weight.
   2. The reported conclusion must be applied to either the net weight or gross weight. The net weight must exclude the weight of any packaging unless an appropriate footnote is used.
   3. A net weight must be documented for each exhibit, except under the following circumstances: (At the analyst’s discretion, a net weight may be documented in these circumstances.)
      a) Exhibits which will not be analyzed
      b) Samples from a larger exhibit submitted to the laboratory for analysis [Customer collected samples from bulk evidence]
c) Exhibits containing only compounds listed in penalty group 1-A

d) Exhibits which are reported using gross weight

4. If the net weight is less than 0.01 grams, it must be recorded as “Trace”.

5. If the source of the net weight is not obvious, document how the net weight was calculated. When multiple items are grouped in multiple weighing events for net weight determination, the items in each group must be documented.

Example: 10 bags with two weighing events may be documented as:

\[
1.50 \text{ g (bags 1-5)} + 1.53 \text{ g (bags 6-10)} = 3.03 \text{ g net weight}
\]

F. Gross Weight

1. A gross weight is defined as the weight of evidence including the weight of the primary containers that are in contact with the evidence.

2. If a gross weight is to be reported instead of a net weight, then documentation is required to demonstrate that the net weight estimated using the largest/heaviest package to obtain a tare would not cause the item to fall below a critical weight as listed in Critical Weights (SD-03-03).

   a) Estimation is not required if:

   i. Samples from a larger exhibit are submitted to the laboratory for analysis (i.e. customer collected the samples from bulk evidence)

   ii. The gross weight of the item(s) is below the lowest penalty.

G. Weighing Events

1. Record the number of weighing events in LIMS so that a weight uncertainty can be calculated. Add all of the weighing events that comprise the weight calculation including dynamic, static, or both.

   a) Dynamic – If a weighing vessel (e.g. weigh paper, weighing boat, etc.) is used, place it on the balance, zero the balance, and add material without removing the vessel. Otherwise, place the item directly on the balance. Each of these is one weighing event.

   b) Static – Tare a weighing vessel, remove it from the balance, fill, and return the vessel to the balance. This is two weighing events.

   c) Complex (similar packaging) – If tare weight estimation is used for multiple packages that are virtually identical (size, thickness, and sticker labels), select the “similar packaging” tab in LIMS and enter the number of packages.

   Note: The number of weighing events will equal the total number of weighing events of the before analysis weight plus one weighing event for the tare packaging (tare weight must be a dynamic weighing event).

H. If the determination of the weighing event(s) is not obvious, document how it was determined. After Analysis Weight (required)

1. No after analysis weight is required for plant material and exhibits greater than one kilogram.

2. If after analysis weight is less than 0.01 grams, “Trace” must be documented.

3. If the entire sample is consumed in analysis, “0” should be documented.
I. The gross inventory weight will be recorded for DPS retained cases of five pounds or more.

J. If the entire evidence exhibit and/or sub-exhibit is consumed during its analysis:
   1. Any remaining analytical sample must be retained with the evidence
   2. Analytical sample disposition must be documented

4.3 Evidence Sampling Techniques

A. In an exhibit, if one or more negative sample(s) is encountered with other samples in containing a controlled substance, or if preliminary testing does not give consistent results, then sub-division will be necessary.

B. All Items
   1. When all items within an evidence exhibit are sampled, each item must be individually confirmed.
   2. No documentation of sampling is necessary.

C. Pharmaceutical Items
   1. For items that have a pharmaceutical reference which are determined to be visually identical and there is no reason to suspect tampering or counterfeiting due to appearance or intact manufacturer seals, then one of the items may be analyzed (report must state the number of items sampled for analysis and the weight of what was sampled).
   2. In certain instances, composite sampling may be necessary to obtain a positive confirmation.
   3. If composite sampling was used for confirmation, it must be stated on the report. Otherwise, no documentation of the sampling plan is necessary.

D. Clandestine Tablets
   1. For tablets that do not appear to be homogeneous or that appear to be clandestinely manufactured, a statistical sampling plan or the non-statistical method (NSM) will be used.
   2. Information about the sampling procedure used will be documented.

E. Non-Statistical Method (NSM)
   1. For items where an inference to a population is not being made, a sufficient number of items may be selected to attain the maximum applicable weight limit as delineated in the Texas Controlled Substances Act.
   2. The selected evidence will be individually confirmed and separately identified as a sub-evidence exhibit with results, number of items, and weight reported.
   3. The remaining evidence not analyzed will be reported with results as “No Analysis” and number of items.
F. Statistical Sampling Plan (SSP)

1. The statistical sampling plan is based on the hypergeometric distribution that will be used to determine the minimum number of items or samples to be tested and individually confirmed in order to prove that either 50%, 75%, or 90% of the total number of items in an evidence exhibit or population will have the reported result with a 95% confidence level.

2. The following table prescribes the minimum number of items randomly selected from a population to be tested.

<table>
<thead>
<tr>
<th>Number of Samples</th>
<th>90%</th>
<th>75%</th>
<th>50%</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6-7</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>8-10</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11-13</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14</td>
<td></td>
<td>7</td>
<td>4</td>
</tr>
<tr>
<td>15-16</td>
<td></td>
<td>12</td>
<td>8</td>
</tr>
<tr>
<td>17</td>
<td></td>
<td>13</td>
<td>8</td>
</tr>
<tr>
<td>18</td>
<td></td>
<td>14</td>
<td>8</td>
</tr>
<tr>
<td>19</td>
<td></td>
<td>15</td>
<td>8</td>
</tr>
<tr>
<td>20-26</td>
<td></td>
<td>16</td>
<td>9</td>
</tr>
<tr>
<td>27</td>
<td></td>
<td>17</td>
<td>9</td>
</tr>
<tr>
<td>28-29</td>
<td></td>
<td>18</td>
<td>9</td>
</tr>
<tr>
<td>30-31</td>
<td></td>
<td>19</td>
<td>9</td>
</tr>
<tr>
<td>32-37</td>
<td></td>
<td>19</td>
<td>9</td>
</tr>
<tr>
<td>38-39</td>
<td></td>
<td>20</td>
<td>9</td>
</tr>
<tr>
<td>40-48</td>
<td></td>
<td>21</td>
<td>10</td>
</tr>
<tr>
<td>49-58</td>
<td></td>
<td>22</td>
<td>10</td>
</tr>
<tr>
<td>59-69</td>
<td></td>
<td>23</td>
<td>10</td>
</tr>
<tr>
<td>70-88</td>
<td></td>
<td>24</td>
<td>10</td>
</tr>
<tr>
<td>89-109</td>
<td></td>
<td>25</td>
<td>10</td>
</tr>
<tr>
<td>110-159</td>
<td></td>
<td>26</td>
<td>10</td>
</tr>
<tr>
<td>160-279</td>
<td></td>
<td>27</td>
<td>11</td>
</tr>
<tr>
<td>280-939</td>
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<td>28</td>
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</tr>
<tr>
<td>940+</td>
<td></td>
<td>29</td>
<td>11</td>
</tr>
</tbody>
</table>
4.4 Basic Analytical Scheme

The basic analytical scheme for the analysis of seized drug evidence consists of sample preparation and extraction or isolation procedures in various combinations with the following tests and instrumentation. The analyst must determine the appropriate sampling techniques, methods of recovery, extraction procedures including derivatization and instrumental analysis to be used for identification of a compound on a case-by-case basis.

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

B. For the identification of an unknown substance, the following are required:

1. At least two positive independent samples. Exceptions are as follows:
   a) If there is insufficient material for two separate samples, more than one test may be performed using the same sample. A method blank must be performed prior to running the sample.
   b) Independent samples are not required for marihuana identification.
   c) When pharmaceutical identification is used as a positive test.

2. One positive Category A technique with at least one other positive technique from Category B, C, or a different Category A technique that supports the identification.

3. When a Category A technique is not used, at least three different techniques must be utilized that support the identification. Two of these techniques must be from Category B and the third technique can be from either Category B or C and must support the identification.

4. Retention time comparison must be performed using a second independent sample.

5. All test methods that involve the comparison of an unknown to a known require the evaluation of the unknown item(s) to identify characteristics suitable for comparison prior to comparison to known item(s). If a comparison is made, it is considered to have been deemed suitable for comparison.

Table of Categories for Testing from SWGDRUG:

<table>
<thead>
<tr>
<th>Category A</th>
<th>Category B</th>
<th>Category C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Infrared Spectroscopy</td>
<td>Capillary Electrophoresis</td>
<td>Color Tests</td>
</tr>
<tr>
<td>Mass Spectrometry</td>
<td>Gas Chromatography</td>
<td>Pharmaceutical Identifiers</td>
</tr>
<tr>
<td>Raman Spectroscopy</td>
<td>Liquid Chromatography</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ultraviolet Spectroscopy</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Thin Layer Chromatography</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Cannabis only:</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Macroscopic Examination</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Microscopic Examination</td>
<td></td>
</tr>
</tbody>
</table>
C. For the identification of marihuana:
   1. A positive microscopic examination is required.
   2. Microscopic and macroscopic examinations are considered to be different Category B techniques.
   3. For a complete analytical scheme, refer to *Physical Examination of Cannabis sativa L.* (SD-03-05) and *Instrumental Analysis of Cannabis sativa L.* (SD-03-06).

D. Hashish, oil extracts, ashes, charred material, edibles and residues containing visible plant material must be examined microscopically for the physical characteristics of Cannabis sativa L.
   1. If the microscopic examination is positive, proceed with the analysis for the identification of marihuana outlined in *Instrumental Analysis of Cannabis Sativa L.* (SD-03-06).
   2. If the microscopic examination is negative, proceed with the analysis for the identification of unknown substances.

E. Isomer determination is not required for chemicals that are controlled by structural class, where data generated cannot distinguish between isomers that are controlled under the same section of the Health and Safety Code.

F. For dextropropoxyphene, the optical rotation of the sample must be determined by physical test or by information direct from the pharmaceutical manufacturer, Physicians’ Desk Reference, or Diccionario de Especialidades Farmacéuticas.

G. Psilocybin may be identified using LC, TLC and FTIR, or TLC and a derivative procedure on GC/MS.

H. The salt form or base form of the drug will be identified using FTIR or other scientifically accepted procedure.

I. General derivatization not specifically covered in this document will be verified through the use of a positive control (reference standard) analyzed with each case.
   1. The general procedure is as follows:
      a) Extract sample with solvent. Evaporating to dryness may be utilized.
      b) Add derivatizing agent (e.g. BSTFA with TMCS, MSTFA).
      c) Incubate using an appropriate time and temperature.
      d) Analyze derivatized sample.
   2. If an alternate procedure is used, reference the peer reviewed literature in the case record.

J. If the entire evidence exhibit will be consumed during analysis, a method blank must be prepared using the same parameters as the evidence sample and analyzed prior to the evidence sample (e.g., when packaging is rinsed to sample the contents of an exhibit).

K. If a sample contains multiple controlled substances, the analyst must attempt to identify the controlled substance with the highest penalty. Performing additional tests to confirm the presence of additional controlled substances with equal or lesser penalties is done at the discretion of the analyst.
L. Each test will be documented, including extractions and sample preparations used.  
   **Note:** For extraction schemes, “acid” means 0.2 N aqueous sulfuric acid solution, 
   and “base” means 1 M aqueous sodium carbonate solution, unless otherwise 
   documented in the case record.

M. If more than one drug is indicated in a sample, a standard may be analyzed and retention 
   time used to confirm the presence of any substance in the sample.

4.5 **Concluding Examination and Return of the Evidence**

A. All original exhibits should be re-packaged in the original container, if possible. The evidence 
   should be re-sealed in a manner that ensure the integrity of the samples and in a way that 
   would detect tampering.

B. If samples are taken from an exhibit for preservation, the samples will be packaged 
   appropriately and sealed in a manner that would detect tampering.

C. The evidence shall be transferred to the evidence custodian or a secure evidence storage 
   area until its final disposition.

5  **Records**

Laboratory Submission Form (LAB-201)
Seized Drugs Worksheet (LAB-SD-01) or LIMS equivalent

6  **Literature References and Supporting Documentation**

SWDRUG. Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) 
Recommendations; Version 8.0; SWGDRUG: June 2019; pp 14-19.

Frank, R. S., Hinkley, S. W., Hoffman, C. G. Representative Sampling of Drug Seizures in Multiple 


ASTM Standard E2329-17 Standard Practice for Identification of Seized Drugs
1 Scope
To establish procedures for the use of the Seized Drugs Worksheet.

2 Practices

2.1 General Instructions
Document the examination of substances on the LAB-SD-01 when access to LIMS is not available.

2.2 Case Information

A. Lab Case Number (required) – this is the cross-reference to the laboratory report.
B. Analyst (required) – Initials of the individual who analyzes the case.
C. Date Started (required) – The date evidence is opened for analysis.
D. Date Completed (required) – The date laboratory examinations are completed.
E. Page – required when more than one worksheet is used.
F. Gross Inventory Weight – required when the gross inventory weight is five (5) pounds or more.
G. Complete the Outside container box (required) and the Inside container box (optional) with the appropriate description of the evidence container.
H. Additional Notes (optional)

2.3 Examination Information

A. Evidence Description (required)
   1. Exhibit # – The agency item number listed on the laboratory submission form or the item number generated by LIMS. If individual items on the submission form are not numbered or are numbered incorrectly, the numbering should be resolved. Separate (subdivide) exhibits with multiple items if any of these items appear to be different in content, and analyze as separate exhibits.
   2. # Items – Number of individual items received
   3. # Analyzed – Number of individual items on which examinations were performed
   4. Description of Evidence – Although there is not a specific heading for the description of evidence, the space below the listing of the exhibit # will be used for brief descriptions of each item of evidence.

B. Weights
   1. Balance ID (required if a weight is determined) – the ID of the balance that was used to weigh the evidence
   2. Before Analysis (optional) – the gross weight of the exhibit (includes packaging)
   3. Tare (optional) – weight of packaging
   4. Sampled (required when the single pharmaceutical tablet/capsule sampling technique is used) – the weight of the number of items in a pharmaceutical exhibit which are sampled for analysis
5. Net – when required, the net weight of the substance that shall be reported
6. After Analysis (if required) – the weight or number of items of evidence remaining after analysis

C. Preliminary Examinations
   1. For color tests, record observations including color and number of tests performed, as appropriate for the respective test(s).
      a) A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
      b) A negative (-) notation indicates that there was no reaction.
   2. For other preliminary tests, list the tests that were conducted and their respective results

D. Confirmatory Examinations
   1. For instrumental examinations, record observations such as +, -, performed, number of tests performed, reference used, etc. as appropriate for the respective test(s).
   2. For other confirmatory tests, list the tests that were conducted and their respective results.

E. Conclusions
   Substance Identified (required) – the results of the analysis
### SD-03-03  CRITICAL WEIGHTS

#### 1 Delivery/Manufacturing Weights

<table>
<thead>
<tr>
<th>Penalty Group</th>
<th>Critical Weights</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1 g</td>
</tr>
<tr>
<td>2/2A</td>
<td>1 g</td>
</tr>
<tr>
<td>3/4</td>
<td>28 g</td>
</tr>
<tr>
<td>Marihuana</td>
<td>¼ oz.</td>
</tr>
</tbody>
</table>

#### 2 Possession Weights

<table>
<thead>
<tr>
<th>Penalty Group</th>
<th>Critical Weights</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1 g</td>
</tr>
<tr>
<td>2</td>
<td>1 g</td>
</tr>
<tr>
<td>3/4</td>
<td>28 g</td>
</tr>
<tr>
<td>2A/Marihuana</td>
<td>2 oz</td>
</tr>
</tbody>
</table>
SD-03-04 EXAMINATION AND DESTRUCTION OF EXCESS QUANTITY SEIZED DRUG PROPERTY

1 Scope

Provisions for the examination and destruction of seized drug property or plant material submitted by a DPS officer and/or a DPS Task Force that has been identified as excess quantity in accordance with the Texas Health and Safety Code Subtitle C Chapter 481 Subchapter E §481.160 and Texas DPS Administrative Code Title 37 Part 1 Chapter 13 Subchapter G Section 13.151.

The intent of this policy is to minimize the total volume of controlled substances retained by the Crime Laboratory Service. This policy does not apply to items which are not identified as controlled substances.

2 Safety

A. Use caution when handling any unknown substance or chemical.

B. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.

C. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

D. Use proper lifting techniques and caution when handling heavy items.

E. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

3 Definitions

3.1 Excess Quantity

A. Defined as greater than:

1. 250 grams of bulk packaged marihuana

2. One (1) kilogram of bulk dry evidence, such as powder

3. 500 milliliters of bulk liquid evidence, such as chemical precursors or liquid controlled substances

4. 200 dosage or abuse units of an item, such as tablets, capsules, or liquids

5. Five (5) individual controlled substance plants, such as marihuana or peyote

6. Five (5) miscellaneous items of drug or inhalant paraphernalia

4 Practices

If it is known at time of submission that the case is associated with a federal prosecution, the federal prosecutor must be contacted prior to analysis and reduction. Authorization is needed to follow this policy associated with excess quantity reduction.

4.1 Documentation and Examination

A. All evidence packages must be either marked with the case number upon receipt or segregated and sampled within five business days of receipt.
B. Photograph(s), digital media, or video recording(s) must be taken and preserved that reasonably depicts each individual item of seized drug property or plant material.
   1. Photographs, digital media, and/or video that are representative of excess quantity controlled substances or plant material will be maintained as evidence
   2. Duplicate copies of evidentiary photographs may be prepared for the submitting agency and/or as documentation photographs.

C. Conduct examinations as necessary under Examination of Seized Drug Evidence (SD-03-01).

D. The net weight or volume of each representative sample and excess quantity exemplar (if retained) of seized drug property or plant material shall be recorded in the case record.

E. The gross inventory weight shall be recorded. The excess quantity representative samples and exemplar may be stored in the same container and included in the gross inventory weight.

F. If gross weights or measurements are unable to be obtained (i.e. items associated with manufacturing), the weight or measurement may be estimated and recorded after making dimensional measurements of the total amount.

G. The report will contain the following footnote, informing the customer that part of the submitted evidence will be destroyed:

   "The excess quantity of the drug evidence will be destroyed in accordance with Texas Health and Safety Code 481.160."

4.2 Representative Samples

A minimum of five random and representative samples must be collected and retained from the total amount of seized drug property or plant material.

A. The total weight of the five random and representative samples retained shall add up to at least the excess quantity amount defined.

B. For bulk liquid evidence in a single container, one representative 500 mL sample is sufficient.

C. In the case of illicit chemical laboratories, a sample of reduced size may be retained if it is determined to be material that is too hazardous to handle or safely store.

4.3 Excess Quantity Exemplar

A. An example of the submitted material (which meets or exceeds the defined criteria for excess quantity as one complete package unit) may be optionally retained as an excess quantity exemplar.

B. An example of an excess quantity exemplar would be one bundle or brick.

4.4 Marking for Identification

A. All retained samples shall be individually packaged, properly sealed, and marked for identification with laboratory case numbers and analyst initials.

B. All individually packaged retained samples may be combined into one container for storage purposes.

C. The retained samples shall be stored in a secure location until the disposition of the case and/or evidence.
5 Destruction of the Excess Quantity

A. The seized drug property or plant material which is not being retained as the representative sample and/or exemplar will be destroyed after analysis according to the Destruction of Evidence chapter of the CLS Manual.

Note: The laboratory may retain seized drug property or plant material in amounts greater than the defined excess quantities as necessary.

B. Each individual item (e.g. brick, bundle, etc.) of the excess quantity scheduled for destruction is not considered evidence and does not require labeling with laboratory case numbers, initials, or seals, so long as it is identifiable and/or segregated by case number.

C. Excess quantity scheduled for destruction shall be maintained in a secure location and inventoried prior to destruction.

6 Records

Case Record

Written Authorization for Destruction
SD-03-05 PHYSICAL EXAMINATION OF CANNABIS SATIVA L.

1 Scope
To establish microscopic and macroscopic procedures for the examination of Cannabis sativa L.

2 Related Chapters
Examination of Seized Drug Evidence (SD-03-01)
Instrumental Analysis of Cannabis sativa L. (SD-03-06)

3 Safety
A. The plant material, dust, and mold often present in botanical substances, may trigger allergic reactions, requiring susceptible personnel to take precautionary measures, such as wearing masks, respirators and gloves.
B. Safety Data Sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.
C. Use proper lifting techniques and caution when handling heavy items.
D. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

4 Equipment and Materials
Stereoscope

5 Practices
5.1 Plant Material
A. Macroscopic characteristics:
   1. Leaves that are palmate in shape
   2. Leaflets having serrated edges
   3. Stems that are fluted in appearance and covered in hairs
   4. Flowering tops/buds
   5. Ovoid shaped seeds with a mottled pattern
B. Microscopic characteristics:
   1. Cystolithic hairs (“bear-claw” hairs): Usually white in color and point towards the terminal portion of the leaf. Present on the upper side of the leaf.
   2. Conical trichomes (Filamentous hairs): Usually white in color like cystolithic hairs but are silky in appearance. Present on the lower side of the leaf.
5.2 Sample Analysis

A. A positive microscopic examination is required for the identification of marihuana or Cannabis sativa L.

B. If sufficient physical characteristics are not observed during the microscopic examination, proceed with the basic analytical scheme as described in Examination of Seized Drug Evidence (SD-03-01).

6 Interpretation

A. Macroscopic Observations
   1. A result is considered positive when one or more of the characteristics listed are observed.
   2. A positive macroscopic examination is indicated by a positive notation on the worksheet along with documentation of the specific characteristic(s) observed. A digital image may also be included in the case record.

B. Microscopic Observations
   1. A result is considered positive when cystolithic hairs and conical trichomes on opposite sides of a leaf are observed.
   2. Glandular hairs may also be present on leaf material.
   3. A positive microscopic examination is indicated by a positive notation on the worksheet along with documentation of the specific hairs observed. A digital image may also be included in the case record.

7 Records

Seized Drugs Worksheet (LAB-SD-01) or LIMS equivalent

8 Literature References and Supporting Documentation


SD-03-06 INSTRUMENTAL ANALYSIS OF CANNABIS SATIVA L.

1 Scope
To establish a procedure that is used to distinguish between marihuana and potential hemp, as defined by Section 121.001 of the Agriculture Code. This is accomplished by determining a decision point for $\Delta^9$-tetrahydrocannabinol (THC) at 1% (w/w) for reporting purposes, utilizing an internal standard, $\Delta^9$-tetrahydrocannabinol-d$_3$ (THC-d$_3$).

2 Related Chapters
Reporting Guidelines (SD-01-01)
Case Documentation (SD-01-04)
Reagents (SD-02-04)
Examination of Seized Drugs Evidence (SD-03-01)
Physical Examination of Cannabis sativa L. (SD-03-05)
Gas Chromatography / Mass Spectrometry (GC/MS) (SD-06-03)

3 Safety
A. The plant material, dust, and mold often present in botanical substances, may trigger allergic reactions, requiring personnel to take precautionary measures, such as wearing masks, respirators and gloves.
B. Use proper lifting techniques and caution when handling heavy items.
C. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

4 Equipment and Materials
- Agilent gas chromatograph/mass spectrometer
- Chromatographic column: 5% Phenyl/95% dimethylpolysiloxane, 30m x 0.25mm x 0.25µm
- Analytical balance
- Class A volumetric flasks
- Mechanical pipettes
- Glass test tubes
- Autosampler vials with reduced volume inserts and caps
- Methanol (Analytical grade or higher)
- Purchased reference standards:
  - $\Delta^9$-tetrahydrocannabinol (THC)
  - $\Delta^9$-tetrahydrocannabinol-d$_3$ (THC-d$_3$)
  - Cannabinoid Mixture (must contain $\Delta^9$-tetrahydrocannabinol, cannabidiol, and cannabinol at equal concentrations)
5 Standards, Controls, and Calibration

5.1 Stock Solutions

Stock solutions can be made to any volume as long as the proper proportions are used.

A. THC Standard Stock Solution (0.05 mg/mL)
   1. Use a mechanical pipette to transfer 0.5 mL (500 µL) of the THC standard (1 mg/mL) into a 10 mL Class A volumetric flask.
   2. Dilute the standard up to volume with methanol.
   3. Refrigerate until use.
   4. Reagent expires three months after the date of preparation.

B. Internal Standard Stock Solution (ISS) (0.1 mg/mL)
   1. Use a mechanical pipette to transfer 1 mL of the THC-d₃ standard (1 mg/mL) into a 10 mL Class A volumetric flask.
   2. Dilute the standard up to volume with methanol.
   3. Refrigerate until use.
   4. Reagent expires three months after the date of preparation.

C. Cannabinoid Mixture Stock Solution (0.05 mg/mL)
   1. Use a mechanical pipette to transfer 0.5 mL (500 µL) of the Cannabinoid Mixture standard (1 mg/mL) into a 10 mL Class A volumetric flask.
   2. Dilute the standard up to volume with methanol.
   3. Refrigerate until use.
   4. Reagent expires three months after the date of preparation.

5.2 Controls

A. All controls will be prepared with the batch of unknown samples.

B. Positive Control
   1. Mix equal volumes of the THC Standard Solution with ISS (example: 100 µL each).
   2. Place sample into an autosampler vial containing a low volume insert.

C. Negative Control
   1. Mix equal volumes of the ISS with MeOH (example: 100 µL each).
      a) Must use the same methanol used to prepare all unknown samples
   2. Place sample into an autosampler vial containing a low volume insert.

D. Secondary Control
   1. Mix equal volumes of the Cannabinoid Mixture Stock Solution with ISS (example: 100 µL each).
   2. Place sample into an autosampler vial containing a low volume insert.
6 Procedure

6.1 Sample Preparation

1. Perform a microscopic examination prior to performing this procedure.
   a) If the microscopic examination is positive, proceed with the following analysis.
   b) If the microscopic examination is negative, proceed with the analytical scheme for unknown substances.

2. If there is less than 150 mg of plant material do not proceed with analysis and report as “No Analysis” with the result note “Insufficient sample for identification.”

3. Using an appropriate balance, weigh 50 mg (±0.5 mg) of dry Cannabis sativa L. and transfer into a glass test tube.
   a) Sample from the buds/leaves of the plant.
   b) The plant material may be broken up with gloved fingers, if necessary.

4. Using a mechanical pipette, add 10 mL of methanol to the test tube containing the plant material.

5. Vortex or agitate the sample for approximately 10 seconds.

6. Let the extract stand for approximately 5 minutes.

7. Vortex or agitate the sample for approximately 10 seconds.

8. Transfer extract from the glass test tube to a storage container.
   a) If particulate material is present, the sample extract can be filtered through a Pasteur pipette containing cotton or glass wool, through a syringe filter, or a similar device.
   b) Sample extract can be stored, capped, in the refrigerator for up to 5 days.

9. Mix equal volumes of the unknown sample extract with ISS (example: 100 µL each).
   a) The ISS used to prepare the batch of unknown samples must be the same ISS that was used to prepare all controls.
   b) All samples for the GC/MS (including the controls) must be prepared on the same day.

10. Place sample into an autosampler vial containing a low volume insert.

11. A dilution on the unknown sample extract might be necessary for analysis.
    a) The plant extract will be diluted 5x with MeOH (example: 50 µL plant extract into 200 µL methanol.
    b) Mix equal volumes of the diluted unknown sample extract with ISS (example: 100 µL each).
    c) If the sample was diluted, it must be noted in the case record. This can be recorded on the data or in the worksheet.
6.2 Instrumental Analysis

A. Samples will be analyzed using the CB_LONG acquisition method that was validated for this purpose.

B. CB_LONG Method Parameters (~12 minute run)

- **Inlet Temperature**: 250°C
- **Injection Mode**: Split
- **Split Ratio**: Between 20:1 and 250:1 *
- **Injection volume**: 1 μL
- **Solvent rinse**: Methanol (A&B)
- **Carrier Gas and Flow**: Helium, 1.5 mL/min
- **Control Mode**: Constant flow
- **Oven Program**: 200 °C initial temperature ramped to 235°C at 15 °C /min; hold for 7 min; ramp to 290°C at 30 °C/min; hold for 1 min.
- **Minimum Run Time**: 12.17 min
- **Gain**: 1
- **Dwell**: 40
- **Ionization Mode**: Electron ionization
- **Solvent Delay**: 2 min
- **Scan Range**: 40-550 m/z
- **MS Source Temperature**: 230°C
- **MS Quadruple Temperature**: 150°C
- **Transfer Line Temperature**: 280°C
- **Tune Type**: stune
- **SIM/Scan Acquisition**:

<table>
<thead>
<tr>
<th>Scan</th>
<th>40-550 m/z</th>
</tr>
</thead>
<tbody>
<tr>
<td>SIM (40 ms)</td>
<td>THC: 314, 231, 271</td>
</tr>
</tbody>
</table>

* Each laboratory must validate its own split ratio(s) based on instrument sensitivity

C. CB_LONG requires the instrument to be tuned using Standard Spectra Tune (Stune.U).

1. The following criteria must be met in order for Agilent GC/MS to pass tune:

   a) **Peak widths must be in the range of 0.40 to 0.70 amu.**
   b) **Mass assignments must be within ±0.2 amu for 69.0, 219.0, and 502.0.**
   c) **Relative Abundance should be as follows:**

<table>
<thead>
<tr>
<th>Parent Peak (amu)</th>
<th>Relative Abundance</th>
</tr>
</thead>
<tbody>
<tr>
<td>69.0</td>
<td>100.0%</td>
</tr>
<tr>
<td>219.0</td>
<td>&gt; 30.0%</td>
</tr>
<tr>
<td>502.0</td>
<td>&gt; 1.0%</td>
</tr>
</tbody>
</table>

   d) **Oxygen (32 amu), Nitrogen (28 amu), and water (18 amu) peaks should be < 20% of the 69 peak abundance.**

2. **Instruments using this method shall be tuned at least monthly.**
D. The THC_SCREENmac Macro will be used for data collection and processing. To run the macro, enter "C:\msdchem\THC_SCREENmac\startxda.mac" in the Acquisition field **prior to starting the sequence run**.

1. The macro performs all of the calculations required by the method.
2. If the macro is unavailable, the evaluation of the data must be manually calculated.

![Screen Macro Diagram]

E. Sequence

1. In the sequence table add the keywords to the sample names, as appropriate. These keywords are:
   a) Daily positive
   b) Secondary
   c) Negative
   d) Positive

2. The controls, blanks, and unknown samples will all be injected at 1 µL onto the GC/MS in the following order:
   a) Daily positive control
   b) Secondary control
   c) Negative control
   d) Methanol blank
   e) Unknown plant samples

3. Methanol blanks must be injected between each unknown plant sample and may be run between controls.

4. The positive control, which is also used as the daily positive control, must be re-injected after every 10 unknown plant sample and at the end of the samples.
5. Example template for sequence:

![Sequence Table]

### 6.3 Scan Data Processing

A. Scan data may be used to identify compounds present in the total ion chromatogram (TIC).

B. Follow *Interpretation* guidelines in SD-06-03.

### 6.4 SIM Data Processing

A. Criteria for Identification

1. **Ion Ratios**
   
   a) *The first injection of the daily positive control is used to set the ion ratios for the batch.*

<table>
<thead>
<tr>
<th>Compound</th>
<th>Target Ions</th>
</tr>
</thead>
<tbody>
<tr>
<td>THC</td>
<td>Base Peak: 314, Qualifiers: 231, 271</td>
</tr>
<tr>
<td>THC-d₃</td>
<td>Base Peak: 317, Qualifiers: 234, 274</td>
</tr>
</tbody>
</table>

   b) *The ion ratios of the remaining controls and unknown samples must be within ± 20% of the set point to be considered positive for that compound.*

   i. *This is determined by the software.*

   ii. *If a qualifier ion is out of tolerance, it is marked with a pound sign (#) on the instrument printout.*

2. **Retention Time**

   a) *The first injection of the positive control (daily positive control) is used to set the retention times for THC and THC-d₃ for the batch.*

   b) *The remaining controls and unknown samples must have retention times within 1% of the set retention times.*

   i. *If this value is calculated using the software/macro, the calculation does not need to be documented in the case record.*
ii. If the value is calculated manually, the calculation must be documented in the case record.

c) Analysts are not allowed to re-integrate peaks. The software/macro will perform the integration.

3. Relative Peak Area (RPA)
   a) The selected ion monitoring (SIM) data is used to determine the RPA of THC/THC-d3 for all samples
   b) RPA = Peak area of THC (314 ion) ÷ Peak area of THC-d3 (317 ion)
   c) The positive control with the highest RPA of THC/THC-d3 is used to set the 1% (w/w) decision point.
      i. This value is also used to normalize all of the values for the unknown samples analyzed in one batch.
      ii. Values are normalized by dividing all of the RPAs by the highest RPA

4. Peak Shape
   a) The 317 and 314 ion peaks from the SIM data should appear as a Gaussian peak shape. Examples:
      i. Acceptable peak shape:

ii. Poor peak shape:

![Graph of peak shape comparison]

B. Controls

1. All injections of the positive controls, including the daily positive control, must have the following to be considered passing:
   a) RPA values within ± 20% of the average. For example:
      i. If the average RPA value is 0.5, 20% is equal to 0.1.
      ii. Therefore, all values from 0.4 to 0.6 are within ± 20%.
   b) If the RPA values are not within ± 20% of the average, then the entire batch fails.
   c) Retention time difference between the set point and the control must be within ±1%.
   d) The 317 and 314 ion peaks from the SIM data should appear as a Gaussian peak shape.

2. The secondary control must have the following to be considered passing:
   a) RPA value within ± 20% of the first positive control (daily positive control)
   b) Retention time difference between the set point and the control must be within ±1%.
   c) The 317 and 314 ion peaks from the SIM data should appear as a Gaussian peak shape.

3. The negative control must have the following to be considered passing:
   a) Presence of internal standard
   b) Either no THC present or the peak area for the THC must be 50 or less.

4. All controls run in the batch must be included in the case record for every case run in the batch.

5. If one of the initial controls fail prior to the analysis of unknown samples (daily positive, secondary, or negative), the control that failed may be re-prepared and re-analyzed.
   a) If a control is re-prepared and re-analyzed, all controls will be re-injected to compare. Do not ONLY inject the control that failed.
   b) The failed controls do not need to be retained with the case files unless they were analyzed with unknown samples.
C. Blanks
   1. Blanks are considered acceptable if:
      a) *The ion ratios are out of tolerance, or*
      b) *The peak area for the compound is 50 or less.*
   2. If the blank prior to an unknown sample is not acceptable, the unknown sample must be re-analyzed.

D. Unknown Samples
   1. Determine the RPA of the THC/THC-d$_3$ for the unknown samples.
   2. Compare that value to the positive control used to set the decision point to determine the normalized RPA value:
      \[
      \text{Sample RPA} \div \text{Positive Control with the highest RPA} = \text{Normalized RPA}
      \]
   3. A dilution will be necessary if:
      a) *The ion ratios of THC-d$_3$ are out of tolerance.*
      b) *The ions for THC-d$_3$ have a poor peak shape.*
   4. If normalized RPA is greater or equal to 1:
      a) *The 317 and 314 ion peaks should appear as a Gaussian peak shape.*
      b) *All ion ratios must be within tolerance.*
      c) *Retention time difference between the set point and the unknown sample must be within ±1%.*
   5. If normalized RPA is less than 1:
      a) *The 317 ion peak should appear as a Gaussian peak shape.*
      b) *All THC-d$_3$ ion ratios must be within tolerance.*
      c) *If a dilution was performed on the extract, the extract will need to be reanalyzed without the dilution step.*

7 Interpretation

7.1 Reporting Guidelines
   A. Microscopic examination must be positive for this reporting scheme.
   B. Analysis indicates Marihuana if:
      1. The normalized RPA is greater than or equal to 1, and
      2. All other identification criteria is met.
      3. The substance is reported as “Contains Marihuana.”
      4. For other reporting guidelines for Marihuana, refer to SD-01-01.
C. Analysis indicates Cannabis sativa L. if:
   1. The normalized RPA is less than 1 or invalid due to THC not being identified.
      a) If THC is not identified, another cannabinoid must be identified (for example cannabinol, cannabidiol, cannabigerol).
   2. The substance is reported as "Contains Cannabis sativa L." with the following result note:
      “The concentration of delta-9-tetrahydrocannabinol (THC) was determined to be below the laboratory’s administrative threshold established for the identification of Marihuana.”

D. Analysis is inconclusive if:
   1. Neither THC nor any other cannabinoid is identified.
   2. Sufficient testing was performed to rule out the presence of any other controlled substance(s).
   3. The substance is reported as, “No controlled substance detected.”

E. If another controlled substance with a higher penalty is identified in or on the plant material, the substance with the higher penalty must be attempted to be confirmed and reported.

8 Precautions
A. Sample extract must be removed from the extraction tube immediately following the 2nd 10 second vortex/agitation in order to stop the extraction process.
B. Method may require more frequent inlet maintenance.

9 Limitations
A. This method is not a quantitative analysis, and the actual amount of THC in the sample will not be determined.
B. This method can only be performed on plant material.
C. This method does not determine if the sample is hemp; it only determines if the plant has a THC concentration above the positive control’s decision point.
D. This method does not include a decarboxylation step, therefore the amount of THC identified in the sample is only what is decarboxylated in the injection port or was naturally decarboxylated in the plant.

10 Advantages
A. The Macro will process the data efficiently and consistently, removing the need for manual calculation.
B. The decision point is 3 times above the legal definition of marihuana, which decreases the likelihood of a false identification.

11 Records
THC Standard Stock Solution Preparation Log (LAB-SD-17)
Internal Standard Stock Solution Preparation Log (LAB-SD-18)
Cannabinoid Mixture Stock Solution Preparation Log (LAB-SD-19)
12 Literature References and Supporting Documentation


SD-03-07 PREPARATION AND ANALYSIS OF MESCALINE IN SUSPECTED PEYOTE

1 Scope
To establish a procedure for the analysis of mescaline in suspected peyote.

2 Safety
A. The plant material and the dust and mold often present in botanical samples may trigger allergic reactions, requiring susceptible personnel to take precautionary measures, such as wearing masks, respirators and gloves.
B. Safety Data Sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.
C. Use proper lifting techniques and caution when handling heavy items.
D. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

3 Equipment and Materials
- Sonicator
- Vortex mixer
- Reagents
  - 0.1 N HCl
  - Petroleum ether
  - NaOH
  - CHCl3
  - 1 M Na2CO3
  - 0.2 N H2SO4

4 Practices

4.1 Visual Identification
1. Examine the plant material for the following physical characteristics:
   a) Small, gray-green spineless cactus, approximately 1-3 inches in diameter.
   b) The top of the cactus may consist of discs that bear tufts of yellowish hair, which produces a small white or pink flower.

2. Document the overall observed characteristics of the evidence.

4.2 Sample Preparation
A. Procedure A:
   1. Cut up plant material (approximately one button) and cover with 0.1 N HCl.
   2. Sonicate extract for 15 minutes and then filter.
   3. Wash extract with petroleum ether and discard the organic layer.
   4. Make aqueous layer basic with NaOH and extract with petroleum ether.
5. Extract petroleum ether layer with 0.2 N H₂SO₄. The aqueous layer may be analyzed by UV/VIS.

6. Make aqueous layer basic and extract with chloroform.

7. Concentrate the chloroform layer. The chloroform layer may be analyzed with a confirmatory test (either GC/MS or FTIR).

B. Procedure B:
   1. Cut the plant material into pieces and soak in methanol.
   2. Filter methanol extract. The methanol extract may be analyzed by UV/VIS, TLC, and a confirmatory test (either GC/MS or FTIR).

C. Procedure C:
   1. Cut plant material into pieces and place in a beaker.
   2. Cover with 1 M Na₂CO₃.
   3. Bring the solution to a slow boil for approximately 20 minutes.
   4. Allow the solution to cool and filter.
   5. Make extract acidic.
   6. Wash with CHCl₃ three times and discard CHCl₃.
   7. Make extract basic add CHCl₃ and filter.
   8. The extract may be analyzed by either GC/MS or FTIR.

5 Interpretation
Proceed with desired analytical testing.

6 Literature References and Supporting Documentation
SD-03-08 IDENTIFICATION OF PSILOCIN / PSILOCYBIN

1 Scope
To establish a procedure for the preparation and identification of Psilocin and Psilocybin

2 Safety
A. The plant material, dust, and mold often present in botanical materials may trigger allergic reactions, requiring susceptible personnel to take precautionary measures, such as wearing masks, respirators and gloves.
B. Safety Data Sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.
C. Use proper lifting techniques and caution when handling heavy items.
D. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

3 Equipment and Materials
- Sonicator
- Centrifuge
- Vortex mixer
- Laboratory oven
- Filter (glass wool, filter paper, etc)
- Reagents
  - Methanol
  - 0.1 N Hydrochloric acid (HCl)
  - Diethyl ether
  - Sodium hydroxide (NaOH)
  - Chloroform (CHCl₃)
  - Glacial acetic acid
  - 0.2 N Sulfuric acid (H₂SO₄)
  - Aqueous base solution
  - BSTFA [N,O-bis(trimethylsilyl)trifluoroacetamide]
  - Acetone

4 Practices
4.1 Identification of Psilocin
A. Procedure A:
   1. Soak/sonicate sample in methanol (sample may be ground to increase efficiency).
   2. Filter out the solids and concentrate the methanol extract (~1-2 mL). The methanol extract may be analyzed by color test, TLC, UV/VIS, FTIR, or GC/MS, or continue with next step.
   3. Add approximately 3 mL diethyl ether to the methanol extract, which will cause a precipitate to form. Agitate the sample.
4. Centrifuge and discard the supernatant liquid. Wash the precipitate with approximately 2 mL diethyl ether; centrifuge and discard the supernatant liquid. Continue washing the precipitate with diethyl ether as needed.

5. Cover the precipitate with methanol. The methanol extract may be analyzed by color test, TLC, UV/VIS, FTIR, or GC/MS.

B. Procedure B:
1. Soak/sonicate sample in 0.2 \( \text{N } \text{H}_2\text{SO}_4 \). Soak at least 30 minutes. Sample may be ground to increase efficiency.

2. Filter out the solids, make the acid extract basic and extract with chloroform. The extract may be analyzed by color test, TLC, UV/VIS, FTIR, or GC/MS.

C. Procedure C:
1. Add 15-20 mL \( \text{H}_2\text{O} \) to ground/powder sample.

2. Add 1-2 mL glacial acetic acid and stir.

3. Allow to soak at least 10 minutes.

4. Filter solution to remove plant material.

5. Make solution basic (~pH 10) with NaOH.

6. Extract solution with CHCl\(_3\). The extract may be analyzed by color test, TLC, UV/VIS, FTIR, or GC/MS.

4.2 Identification of Psilocybin

A. Procedure A:
1. Soak sample in methanol overnight. Centrifuge the mixture and collect the supernatant liquid. Methanol extract may be analyzed by color test, TLC, UV/VIS, proceed to derivatization or continue with next step.

2. Add 2-4 mL acetone to the liquid, place in freezer overnight.

3. Centrifuge or filter the mixture and save the liquid.

4. Use a stream of dry air to reduce the volume of the methanol-acetone extract to 0.5-1.0 mL. The methanol-acetone extract may be analyzed by color test, TLC, UV/VIS, FTIR, or proceed to derivatization.

5. Derivatization:
   a) Place a portion of the liquid concentrate into a vial or insert.
   b) Evaporate the liquid extract to dryness with a stream of dry air.
   c) Add approximately 100 \( \mu \text{L} \) BSTFA to the dried residue.
   d) Cap vial and incubate approximately 30 min at 90-100\( ^\circ \text{C} \).

6. Analyze by GC/MS. After the injection of the sample, clean the syringe with ethyl acetate.
B. Procedure B:

1. Soak/sonicate sample in methanol for a minimum of 30 minutes (sample may be ground to increase efficiency).
2. Filter out the solids and concentrate (~1-2 mL). The methanol extract may be analyzed by color test, TLC, UV/VIS, proceed to derivatization, or continue with next step.
3. Add approximately 3 mL diethyl ether to the methanol extract, which will cause a precipitate to form. Agitate the sample.
4. Centrifuge and discard the supernatant liquid. Wash the precipitate with approximately 2 mL diethyl ether; centrifuge and discard the supernatant liquid. Continue washing the precipitate with diethyl ether as needed.
5. Cover the precipitate with methanol. The methanol extract may be analyzed by color test, TLC, UV/VIS, FTIR, or proceed to derivatization.
6. Derivatization:
   a) Place a portion of the methanol extract into a vial or insert.
   b) Evaporate the methanol extract to dryness with a stream of dry air.
   c) Add approximately 100 μL BSTFA to the dried residue.
   d) Cap vial and incubate approximately 30 minutes at 90-100°C.
7. Analyze by GC/MS. After the injection of the sample, clean the syringe with ethyl acetate.

5 Limitations

A. Psilocin and bufotenine are positional isomers and have similar analytical results.
B. Psilocybin dephosphorylates into Psilocin when subjected to heat or pH-shifts; therefore, derivatization is required when using GC/MS to identify Psilocybin.

6 Literature References and Supporting Documentation


SD-03-09 DERIVATIZATION OF LSD

1 Scope
To establish a procedure for preparation and analysis of LSD samples.

2 Safety
A. Safety Data Sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.
B. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

3 Equipment and Materials
- Sonicator
- Centrifuge
- Vortex mixer
- Laboratory oven
- Autosampler vials (or similar) with screw caps
- Reagents
  - 0.2 N H₂SO₄
  - Conc. aqueous NaOH solution
  - BSTFA [N,O-bis(trimethylsilyl)trifluoroacetamide]
  - Chloroform
  - Ethyl acetate

4 Procedure
1. Place between one half and three abuse units in dilute aqueous acid for at least 15 minutes but no more than 12 hours. More units may be used, as determined to be necessary. Sonication may be used to speed the extraction. The acid extract may be analyzed by UV/VIS.
2. Make the extract basic (using conc. NaOH) and extract with chloroform (CHCl₃). The extract may be analyzed by TLC.
3. Evaporate the CHCl₃ solution to dryness using a current of dry air or an oven.
4. Add 1-2 drops BSTFA to the dried residue.
5. Cap vial and incubate approximately 30 min at 90 - 100 °C.
6. Analyze by GC/MS. After the injection of the sample, clean the syringe with ethyl acetate.

5 Interpretation
Proceed with desired analytical testing.

6 Literature References and Supporting Documentation
Harper, Charles W., “Silylation and Acylation Derivatives for GLC and GLC-MS Drug Analysis”, Microgram, Volume XII, No. 4, p 82-86.
SD-03-10 DERIVATIZATION OF GHB

1 Scope
To establish a procedure for preparation and analysis of GHB samples.

2 Safety
A. Use caution when handling any unknown substance or chemical.
B. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
C. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.
D. Use proper lifting techniques and caution when handling heavy items.
E. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

3 Equipment and Materials
- Gas Chromatograph Mass Spectrometer
- Autosampler vials (or similar) with screw caps
- Hot plate or oven (temperature 80 – 90°C)
- Reagents
  - N,O-bis(Trimethylsilyl)-trifluoroacetamide (BSTFA) (Pierce No. 38830 or equivalent) with 1% Trimethylchlorosilane (TMCS) or
  - BSTFA with 1% TMCS (Pierce No. 38831 or equivalent)
  - Acetonitrile
  - Hexane

4 Procedure
1. Place sample of suspected GHB in test tube (approx. 20-50 mg).
2. Allow sample to dry. You may place sample in oven for 1 hour or place on a hot plate.
   Note: You may substitute BSTFA with TMCS for steps 3 and 4.
3. Add 5 drops of BSTFA using Pasteur pipette (1 drop = 10 µL)
4. Add 1 drop of TMCS to test tube with sample and BSTFA reagent.
5. Place test tube in oven for approx. 10 minutes.
6. Remove from oven and let test tube cool.
7. Add 5 drops of Acetonitrile.
8. Add 1 mL Hexane.
9. Remove hexane layer (top) and prepare sample for GC/MS analysis as usual.
5 Interpretation

A. Proceed to GC/MS analysis.

B. The trimethysilyl derivative of GHB is obtained. Gamma butyrolactone does not form a TMS derivative.

6 Literature References and Supporting Documentation

Busby, Claudia, *GHB Analysis Techniques for GHB and 1,4 Butanediol*


04 CHEMICAL SPOT TESTS
SD-04-01 CHEMICAL SCREENING SPOT TESTS – OVERVIEW

1 Scope
To describe the chemical screening procedures, commonly referred to as chemical spot tests, for preliminary testing of seized drug evidence.

2 Safety
A. Chemical spot tests may use a variety of corrosive, caustic, or other dangerous chemicals. Caution should always be practiced, and appropriate personal protective equipment used.
B. Refer to SDS for additional safety information for specific chemicals.

3 Equipment and Materials
- Spot plates, pipettes, or other appropriate containers/items
- Reagents appropriate to the specific chemical spot tests.

4 Standards, Controls, and Calibration
A. Prepared reagents will be labeled with the identity of the reagent, the preparer’s initials, and the date of preparation or lot number.
   1. Records shall be maintained identifying who made the reagent and the components used in preparation.
   2. The analyst’s initials and the date prepared must be recorded in the corresponding reagent preparation/quality control log.
B. Freshly prepared spot test reagents will be performance checked with a positive and negative control and the results recorded in the corresponding log.
C. Unless otherwise specified, performance of reagents will be checked every month and the results of the checks documented in the corresponding log. If the reagent has not been used for a month or more, it must be checked using a standard (and the results of the check logged) before its use with case samples.
D. It is the responsibility of the analyst to determine if reagents are working properly, and to periodically quality test them and document the results. Reagents which do not respond appropriately to quality testing will be discarded.

5 Limitations
A. All spot tests are presumptive in nature and serve only as a guide for an analyst’s analytical scheme.
B. Adulterants and complex mixtures may produce reactions that interfere with the interpretations.

6 Advantages
A. Spot tests provide a quick and easy method for determining what a sample might contain.
B. Spot tests may be used as an indication for more than one drug.
C. Spot tests can assist in the determination of appropriate analytical processing, collection of appropriate samples, and grouping samples for uniformity testing.
7 Records

Corresponding reagent preparation/quality control log
SD-04-02 MARQUIS TEST

1 Scope
To establish test procedures for the presumptive detection of a range of compounds using the Marquis test.

2 Safety
A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials
3.1 Chemicals
- Concentrated sulfuric acid (conc. H₂SO₄)
- Formaldehyde solution (approx. 37% Formaldehyde)

3.2 Reagent Preparation
Add 1 mL formaldehyde solution to 9 mL conc. H₂SO₄ or 1 drop of formaldehyde solution to 1 mL conc. H₂SO₄.

4 Procedure
1. Combine a small amount of sample with a few drops of Marquis reagent.
2. Record any observations.

5 Interpretation
A. The color which appears must be documented on the examination worksheet.
   1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
   2. A negative (-) notation indicates that there was no reaction.
B. Various colors may be produced by a large number of compounds.
   1. A reaction which forms an orange color indicates the possible presence of amphetamine or methamphetamine.
   2. A reaction which forms a black color indicates the possible presence of dextromethorphan, MDA, or one of its analogues.
   3. A reaction which forms a dark purple color indicates the possible presence of heroin, opiates, methocarbamol, or guaifenesin.
   4. A reaction which forms a red color indicates the possible presence of salicylates.
   5. A reaction which forms a bright yellow color indicates the possible presence of methylone, butylone, pentylone, MDPV, or diphenhydramine.
C. Additional results or interpretations may be found in Clarke’s Isolation and Identification of Drugs, Clarke’s Analysis of Drugs and Poisons, electronic DPS Chemical Library and/or additional peer reviewed literature not in the DPS Chemical Library. Peer reviewed literature not in the DPS Chemical Library must be cited in the case notes.
6 Records
Marquis Reagent Preparation/Quality Control Log (LAB-SD-14)

7 Literature References and Supporting Documentation


SD-04-03 SCOTT TEST / COBALT THIOCYANATE TEST

1 Scope
To establish test procedures for the presumptive detection of cocaine base and cocaine salts with the Scott or Cobalt Thiocyanate test.

2 Safety
A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials
3.1 Chemicals
- Cobalt thiocyanate (Co(SCN)2)
- Glycerin
- Purified water (H2O)
- Concentrated hydrochloric acid (HCl) or other acid
- Chloroform (CHCl3)

3.2 Reagent Preparation
A. Scott Reagent
Dissolve 2 g cobalt thiocyanate in 100 mL H2O and dilute with 100 mL glycerin.
B. 2% Cobalt Thiocyanate with Glycerin Reagent
Dissolve 2 g cobalt thiocyanate in 50 mL H2O and dilute with 50 mL glycerin.
C. 2% Cobalt Thiocyanate (Co(SCN)2) Reagent
Dissolve 2 g cobalt thiocyanate in 100 mL H2O.

4 Procedure
4.1 Scott Reagent and 2% Cobalt Thiocyanate with Glycerin Reagent Procedure
1. Combine a small amount of sample with approximately 5 drops of the Reagent. If a blue color is observed, the analyst may stop.
2. Record any observations.
3. Add approximately 1-2 drops of acid to the sample. If a blue color is observed, the analyst may stop.
4. Record any observations.
5. Add approximately five drops of CHCl3 to the sample.
6. Record any observations.
4.2 2% Cobalt Thiocyanate (Co(SCN)₂) Reagent Procedure

1. Combine a small amount of sample with a few drops of the 2% Co(SCN)₂ Reagent. If a blue color is observed, the analyst may stop.

2. Record any observations.

3. Add approximately 1-2 drops of acid to the sample. If a blue color is observed, the analyst may stop.

4. Record any observations.

5 Interpretation

A. The color that appears for each step performed must be documented on the examination worksheet. If more than one step was performed, document the actual colors observed separated by a “/” or “>.”

1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.

2. A negative (-) notation indicates that there was no reaction for all steps.

B. If the addition of the reagent results in a blue color, the addition of acid results in a pink color, and the addition of CHCl₃ results in a blue color in the bottom layer, a cocaine salt may be present.

C. If the addition of the reagent results in no color change or a pink color, the addition of acid results in a blue color, and the addition of CHCl₃ results in a blue color in the bottom layer, cocaine base may be present.

6 Records

2% Cobalt Thiocyanate with Glycerin Reagent Preparation/Quality Control Log (LAB-SD-04A)

2% Cobalt Thiocyanate Reagent Preparation/Quality Control Log (LAB-SD-04B)

Scott Reagent Preparation/Quality Control Log (LAB-SD-04C)

7 Literature References and Supporting Documentation


SD-04-04 MODIFIED SODIUM NITROPRUSSIDE (SNP) TEST

1 Scope
To establish test procedures for the presumptive detection of secondary amines with the modified Sodium Nitroprusside test.

2 Safety
A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials
3.1 Reagents/Chemicals
- Sodium Nitroprusside
- Purified water (H₂O)
- Acetaldehyde
- 1M Sodium Hydroxide (NaOH)
- 1M Sodium Carbonate (Na₂CO₃)

3.2 Reagent Preparation
Dissolve 1 g Sodium Nitroprusside in a mixture of 10 mL Acetaldehyde and 100 mL H₂O.

4 Procedure
1. Either:
   a) Combine a small amount of sample with a few drops of the modified SNP reagent, then add a few drops of 1M Na₂CO₃ (or 1M NaOH) to the sample; or
   b) Combine a few drops of the modified SNP reagent with a few drops of 1M Na₂CO₃ (or 1M NaOH), then add a small amount of sample.
2. Record any observations.

5 Interpretation
A. The color which appears must be documented on the examination worksheet.
   1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
   2. A negative (-) notation indicates that there was no reaction.
B. A reaction that forms a blue color indicates the possible presence of secondary amines, such as methamphetamine.

6 Records
Sodium Nitroprusside (SNP) Reagent Preparation/Quality Control Log (LAB-SD-16)

7 Literature References and Supporting Documentation
SD-04-05  FERRIC CHLORIDE TEST

1  Scope
To establish test procedures for the presumptive detection of a range of compounds using the Ferric Chloride test.

2  Safety
   A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
   B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3  Equipment and Materials
3.1  Chemicals
   • Ferric chloride hexahydrate (FeCl₃•6H₂O)
   • Purified water (H₂O)

3.2  Reagent Preparation
Dissolve 0.83 g FeCl₃•6H₂O in 10 mL H₂O.

4  Procedure
   1. Combine a small amount of sample and a few drops of Ferric Chloride reagent.
   2. Record any observations.

5  Interpretation
   A. The color which appears must be documented on the examination worksheet.
      1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
      2. A negative (-) notation indicates that there was no reaction.
   B. A reaction that forms an orange-brown color indicates the possible presence of GHB.
   C. A reaction that forms a dark purple color indicates the possible presence of salicylates.
   D. A reaction that forms a bluish-gray color indicates the possible presence of acetaminophen.

6  Records
Ferric Chloride Reagent Preparation/Quality Control Log (LAB-SD-09)

7  Literature References and Supporting Documentation
SD-04-06 COBALT NITRATE TEST

1 Scope
To establish test procedures for the presumptive detection of gamma-hydroxybutyrate (GHB) and barbiturates using the Cobalt Nitrate test.

2 Safety
A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials
3.1 Chemicals
- Cobalt nitrate (Co(NO₃)₂)
- Isopropylamine 95% ethanol

3.2 Reagent Preparation
A. 1% Cobalt nitrate reagent:
   Add 1 g cobalt nitrate to 100 mL ethanol.
B. 5% Isopropylamine reagent:
   Add 5 g isopropylamine to 100 mL ethanol

4 Procedure
1. Combine a small amount of sample and a few drops of the 1% cobalt nitrate reagent.
2. Record any observations.
3. Add a few drops of 5% isopropylamine to the sample.
4. Record any observations.

5 Interpretation
A. The color that appears for each step performed must be documented on the examination worksheet. If more than one step was performed, document the actual colors observed separated by a “/” or “>.”
   1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
   2. A negative (-) notation indicates that there was no reaction.
B. A purple color after the addition of the 1% cobalt nitrate reagent indicates the possible presence of gamma-hydroxybutyrate (GHB).
C. A purple color after the addition of the 5% isopropylamine reagent indicates the possible presence of barbiturates.
6  Records

Cobalt Nitrate Reagent Preparation/Quality Control Log (LAB-SD-06)

7  Literature References and Supporting Documentation


SD-04-07  P-DIMETHYLAMINOBENZALDEHYDE (P-DMABA) TEST

1 Scope
To establish test procedures for the presumptive detection of a range of compounds using the p-Dimethylaminobenzaldehyde test.

2 Safety
   A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
   B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials
3.1 Chemicals
   - 95% Ethanol
   - p-Dimethylaminobenzaldehyde (p-DMABA)
   - Concentrated hydrochloric acid (Conc. HCl)

3.2 Reagent Preparation
Dissolve 2 g p-dimethylaminobenzaldehyde in 50 mL ethanol and 50 mL conc. HCl.

4 Procedure
   1. Combine a small amount of sample and a few drops of p-DMABA reagent.
   2. Record any observations.

5 Interpretation
   A. The color which appears must be documented on the examination worksheet.
      1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
      2. A negative (-) notation indicates that there was no reaction.
   B. A reaction which forms a bright yellow color indicates the possible presence of procaine or benzocaine.
   C. A reaction which forms a purple color indicates the possible presence of LSD.

6 Records
p-DMABA Reagent Preparation/Quality Control Log (LAB-SD-15)

7 Literature References and Supporting Documentation
SD-04-08 WEBER TEST

1 Scope
To establish test procedures for the presumptive detection of psilocin using the Weber test.

2 Safety
   A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
   B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials

3.1 Chemicals
   - Fast Blue B
   - Concentrated hydrochloric acid (conc. HCl)
   - Purified water (H₂O)

3.2 Reagent Preparation
Dissolve 0.01 g Fast Blue B in 10 mL H₂O.
*Prepare this reagent fresh before use*

4 Procedure
   1. Combine a small amount of sample (or a methanol extract of the sample) with a few drops of the Weber reagent wait approximately one minute.
   2. Record any observations.
   3. Add one to two drops of conc. HCl.
   4. Record any observations.

5 Interpretation
   A. The color that appears for each step must be documented on the examination worksheet. Document the actual colors observed separated by a “/” or “>.”
      1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
      2. A negative (-) notation indicates that there was no reaction.
   B. If the addition of the Weber reagent results in a red color and the addition of concentrated HCl results in a blue color, psilocin may be present.

6 Literature References and Supporting Documentation
SD-04-09 DUQUENOIS-LEVINE TEST

1 Scope
To establish test procedures for the presumptive detection of marihuana and THC using the Duquenois-Levine test.

2 Safety
A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials

3.1 Chemicals
- Vanillin
- At least 90% Ethanol
- Acetaldehyde
- Concentrated hydrochloric acid (conc. HCl)
- Chloroform (CHCl₃)
- Petroleum ether

3.2 Reagent Preparation
Add 0.4 g vanillin and 5 drops acetaldehyde to 20 mL ≥90% ethanol.

4 Procedure
1. Place a small amount of sample in a testing container.
   a) Either proceed directly to the next step or extract the sample with petroleum ether or another suitable solvent.
   b) If extracted, discard the insoluble material, and evaporate the extract to dryness.
2. Add one volume of the Duquenois reagent and wait approximately one minute. It is not necessary to wait as long when using the extract.
3. Add one volume of conc. HCl.
4. Add one volume of CHCl₃.
5. Record any observations.

5 Interpretation
A. The color which appears after the addition of CHCl₃ must be documented on the examination worksheet.
   1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
   2. A negative (-) notation indicates that there was no reaction.
B. A purple color in the CHCl₃ layer indicates that the components (cannabinoids, including THC) unique to Cannabis, marihuana, or hashish are present.
6 Records
Duquenois Reagent Preparation/Quality Control Log (LAB-SD-07)

7 Literature References and Supporting Documentation

SD-04-10 FORMALDEHYDE-SULFURIC ACID TEST

1 Scope
To establish test procedures for the presumptive detection of benzodiazepines using the Formaldehyde-Sulfuric Acid test.

2 Safety
A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials

3.1 Chemicals
- Concentrated sulfuric acid (conc. H₂SO₄)
- Formaldehyde solution (i.e. 37% Formaldehyde)

3.2 Reagent Preparation
Add 6 volumes of formaldehyde solution to 4 volumes conc. H₂SO₄.

4 Procedure
1. Combine a small amount of sample and a few drops of formaldehyde-sulfuric reagent in a test tube or spot plate.
2. Heat at 100 °C for approximately one minute.
3. Record any observations.

5 Interpretation
A. The color which appears must be documented on the examination worksheet.
   1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
   2. A negative (-) notation indicates that there was no reaction.
B. Benzodiazepines generally will produce an orange color.

6 Records
Formaldehyde Sulfuric Test Reagent Preparation/Quality Control Log (LAB-SD-10)

7 Literature References and Supporting Documentation
SD-04-11 LIEBERMANN TEST

1 Scope
To establish test procedures for the presumptive detection of a range of compounds using the Liebermann Test.

2 Safety
A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials
3.1 Chemicals
- Sodium nitrite (NaNO₂)
- Sulfuric acid (H₂SO₄)

3.2 Reagent Preparation
Add 5 g NaNO₂ to 50 mL H₂SO₄ with cooling and swirling in the hood, as toxic nitrogen oxides are produced.

4 Procedure
1. Combine a small amount of sample and a few drops of Liebermann reagent.
2. If required by literature, heat at 100 °C for approximately one minute.
3. Record any observations.

5 Interpretation
A. The color which appears must be documented on the examination worksheet.
   1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
   2. A negative (-) notation indicates that there was no reaction.
B. Various colors may be produced by a large number of different compounds. Additional results or interpretations may be found in Clarke’s Isolation and Identification of Drugs, Clarke’s Analysis of Drugs and Poisons, Poyner & Morris (2012), the electronic DPS Chemical Library, and/or additional peer reviewed literature not in the DPS Chemical Library.
C. Peer reviewed literature, not in the DPS Chemical Library, must be cited in the case notes.

6 Records
Liebermann Test Reagent Preparation/ Quality Control Log (LAB-SD-12)

7 Literature References and Supporting Documentation
SD-04-12 JANOVSKY TEST

1 Scope
To establish test procedures for the presumptive detection of ketamine and/or flunitrazepam using the Janovsky Test.

2 Safety
A. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
B. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

3 Equipment and Materials

3.1 Chemicals
- m-dinitrobenzene
- Potassium hydroxide (KOH)
- Absolute ethanol
- Purified water (H$_2$O)

3.2 Reagent Preparation
1. Janovsky Solution A
   2% m-dinitrobenzene: Add 2 g m-dinitrobenzene to 100 mL absolute ethanol.
2. Janovsky Solution B
   5 N potassium hydroxide: Add 28.05 g KOH to 100 mL H$_2$O.
3. Combine equal amounts of Janovsky Solution A and B in an appropriate container.

4 Procedure
1. Add a small amount of sample to the Janovsky reagent.
2. Heat for approximately 3-5 seconds.
3. Record any observations.

5 Interpretation
A. The color which appears must be documented on the examination worksheet.
   1. A positive (+) notation may be added to the observation to indicate that the test was used to support the conclusion.
   2. A negative (-) notation indicates that there was no reaction.
B. A reaction which forms an initial brown/purple color with purple precipitate or specks indicates the possible presence of Ketamine. The purple color will intensify with heat and time.
C. A reaction which forms an initial strong purple color and fades to brown indicates the possible presence of Flunitrazepam.
6 Records

Janovsky Test Reagent Preparation/Quality Control Log (LAB-SD-11)

7 Literature References and Supporting Documentation


05  THIN-LAYER CHROMATOGRAPHY
SD-05-01  THIN-LAYER CHROMATOGRAPHY

1  Scope
To outline procedures for the use of thin-layer chromatography as an analytical method.

2  Related Chapters
Marquis Test (SD-04-02)
p-Dimethylaminobenzaldehyde (p-DMABA) Test (SD-04-07)

3  Safety
A. Use appropriate eye protection, gloves and lab coat to avoid any contact with the chemicals that are involved with this technique. This technique should be performed in a fume hood.
B. Care should be used when spraying the TLC plates to avoid accidental ingestion of the reagent or exposure of the skin and eyes to the reagent. Refer to the appropriate SDS for the safe handling of the solvents and reagents used in this technique.
C. Developing solvents and indicator reagents should be discarded in an appropriate manner.

4  Equipment and Materials

4.1  Equipment/Materials
- Silica gel thin-layer chromatography plates
- Developing chamber
- Micropipettes (1-5 µL) or equivalent
- UV light box (long and short wave)

4.2  Reagents
A. All reagents will have an expiration date of two years from the date of preparation.
B. Reagents should be stored following manufacturer guidelines.
C. Approved TLC solvent systems

<table>
<thead>
<tr>
<th>System ID</th>
<th>Solvent System (ratios of respective solvents)</th>
<th>Typical Drugs Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>50:25:15:10 (cyclohexane:toluene:acetone:diethylamine)</td>
<td>Cannabis sativa L.</td>
</tr>
<tr>
<td></td>
<td>The extraction solvent used to prepare samples will be petroleum ether, unless otherwise noted.</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>18:1 (chloroform, sat. with ammonia:methanol)</td>
<td>LSD</td>
</tr>
<tr>
<td>3</td>
<td>9:2 (chloroform:methanol)</td>
<td>LSD</td>
</tr>
<tr>
<td>4</td>
<td>9:1 (acetone:chloroform sat. with ammonia)</td>
<td>LSD</td>
</tr>
<tr>
<td>5</td>
<td>2:1:1 (n-butanol:acetic acid:water)</td>
<td>Psilocybin</td>
</tr>
<tr>
<td>6</td>
<td>TA 1.5:100 (ammonium hydroxide:methanol)</td>
<td>General</td>
</tr>
<tr>
<td>7</td>
<td>TB 75:15:10 (cyclohexane:toluene:diethylamine)</td>
<td>General</td>
</tr>
<tr>
<td>8</td>
<td>TC 90:10 (chloroform:methanol)</td>
<td>General</td>
</tr>
<tr>
<td>9</td>
<td>TL (acetone)</td>
<td>General</td>
</tr>
<tr>
<td>System ID</td>
<td>Solvent System (ratios of respective solvents)</td>
<td>Typical Drugs Analyzed</td>
</tr>
<tr>
<td>-----------</td>
<td>-----------------------------------------------</td>
<td>------------------------</td>
</tr>
<tr>
<td>10</td>
<td>TAE (methanol)</td>
<td>General</td>
</tr>
<tr>
<td>11</td>
<td>TAF 60:40 (Methanol: n-butanol) and 0.1mol/L NaBr</td>
<td>General</td>
</tr>
<tr>
<td>12</td>
<td>Davidow 85:10:5 (ethyl acetate:methanol:ammonium hydroxide)</td>
<td>General</td>
</tr>
<tr>
<td>13</td>
<td>13:1.9: 0.1 (methyl ethyl ketone: dimethylformamide: NH₄OH)</td>
<td>Mescaline</td>
</tr>
<tr>
<td>14</td>
<td>1:1 (ethyl acetate:hexanes)</td>
<td>Salvinorin A</td>
</tr>
</tbody>
</table>

D. Approved indicating reagents

<table>
<thead>
<tr>
<th>Indicating Reagents</th>
<th>Typical Drugs Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fast Blue RR</td>
<td>Cannabis sativa L. and THC</td>
</tr>
<tr>
<td>p-DMABA (SD-04-07)</td>
<td>LSD, Psilocybin mushrooms, and indoles</td>
</tr>
<tr>
<td>Ninhydrin</td>
<td>Mescaline and Amines</td>
</tr>
<tr>
<td>Acidified Iodoplatinate</td>
<td>Mescaline, Opiates, and tertiary amines</td>
</tr>
<tr>
<td>Marquis (SD-04-02)</td>
<td>General substances</td>
</tr>
<tr>
<td>Potassium permanganate</td>
<td>General substances</td>
</tr>
<tr>
<td>Vanillin Reagent</td>
<td>Salvinorin A</td>
</tr>
</tbody>
</table>

E. Preparation of select indicating reagents

1. **Fast Blue RR reagent:**
   a) **Chemicals**
      i. Purified H₂O, methanol, or ethanol
      ii. Fast Blue RR salt
   b) **Dissolve 0.25 g Fast Blue RR salt in 50 mL solvent.**
   c) **Developed spot for THC appears red.**

2. **Ninhydrin reagent:**
   a) **Chemicals**
      i. Ninhydrin
      ii. Acetone
   b) **Dissolve 0.5 g ninhydrin in 100 mL acetone.**
   c) **Developed spots appear red to purple.**

3. **Acidified Iodoplatinate reagent:**
   a) **Chemicals**
      i. 10% Platinic chloride solution
      ii. 4% Potassium iodide solution
      iii. Purified H₂O
      iv. Concentrated (37%) HCl
b) Mix 5 mL 10% platinic chloride solution with 125 mL 4% KI solution.

c) Dilute to 250 mL with purified water.

d) Add 12.5 mL conc. HCl.

e) Developed spots appear purple or blue.

4. **Potassium permanganate reagent (1%)**:  
   a) **Chemicals**  
      i. 0.5 N sulfuric acid  
      ii. Potassium permanganate (KMnO₄)  
   b) Add 1 g potassium permanganate to 100 mL 0.5 N sulfuric acid.  
   c) Developed spots appear lighter than the background.

5. **Vanillin reagent**:  
   a) **Chemicals**  
      i. 50 mL ethanol  
      ii. 0.3 mL conc. sulfuric acid  
      iii. 1 g vanillin  
   b) Add 1 g vanillin to 50 mL ethanol.  
   c) Add 0.3 mL conc. sulfuric acid.  
   d) Developed spots appear pinkish-purple after heating.

5 Standards, Controls, and Calibration  
An appropriate known reference standard will be used to test the system and indicating reagents.

   A. A standard will be analyzed on all plates.
   B. If the expected result of the standard is not obtained, the cause of the issue will be identified and resolved before the analysis is repeated.
   C. A solvent blank will be run on all plates.
   D. If the expected result of the solvent blank is not obtained, the cause of the issue will be identified and resolved before the analysis is repeated.

6 Procedure  
1. Extract the sample with an appropriate solvent.
2. Spot a suitable amount of extract from the sample and at least one standard on the TLC plate approximately 1.5 cm above the bottom of the plate.
3. Allow the sample to dry after application.
4. Place the plate vertically into a developing chamber with enough solvent mixture to cover 0.5 to 1.0 cm of the sample-end of the plate.
5. Allow the solvent front to rise near the top of the TLC plate.
6. Remove the plate from the solvent and allow it to air dry. Systems containing ammonia may be gently heated to remove the excess ammonia before spraying.

7. Apply an appropriate indicator spray and/or view under UV light to visualize the component(s) of interest.

8. Compare the migration of the sample spot to that of the standard.

9. Document the solvent or extraction procedure used to prepare the samples, the solvent system used to analyze the samples, and the results of analysis.

7 Interpretation

A. A positive determination is made when the spot(s) of the unknown substance matches the color and migration of the spot of the standard.

B. In the absence of reviewable instrumental data, a digital image or color photocopy of the TLC plate must be retained in the case record.

8 Limitations

A. TLC is not considered a confirmatory test and further analysis is necessary for the positive identification of an unknown substance.

B. Various factors limit the determination of $R_f$ values in TLC analysis, including the length of the plate, bleeding of the sample, temperature and developing time. However, the use of multiple systems and chemical locating reagents make it a more specific technique.

9 Advantages

A. Relatively quick and easy technique

B. Can be used as a clean-up procedure for complex mixtures

C. Requires no expensive instrumentation

10 Records

Fast Blue RR TLC Indicator Preparation/Quality Control Log (LAB-SD-08)

Cannabis sativa L. Thin Layer Solvent System Preparation/Quality Control Log (LAB-SD-13)

Acidified Iodoplatinate TLC Indicator Preparation/Quality Control Log (LAB-SD-27)

Ninhydrin TLC Indicator Preparation/Quality Control Log (LAB-SD-28)

Potassium Permanganate TLC Indicator Preparation/Quality Control Log (LAB-SD-29)

11 Literature References and Supporting Documentation


06 INSTRUMENTAL ANALYSIS

SD-06-01 UV/VIS SPECTROPHOTOMETRY

1 Scope

A nondestructive analytical technique for the preliminary identification of seized drug evidence. Ultraviolet/visible spectrophotometers are considered significant equipment for the Seized Drug discipline.

2 Safety

A. Use appropriate safety equipment when preparing reagents and pouring liquids.
B. Refer to the SDS for additional safety information for specific chemicals.

3 Equipment and Materials

- UV/visible spectrophotometer
- Quartz cuvettes, matched pair or equivalent
- An appropriate solution for the sample:
  - Acidic solutions, such as 0.2 N H₂SO₄ or 0.1 N HCl
  - Basic solutions, such as concentrated NaOH or 1.0 M Na₂CO₃
  - Methanol or ethanol

4 Standards, Controls, and Calibration

4.1 Performance Verification/Check

A. Conduct a performance check on UV/VIS instruments at least once each quarter of the calendar year.
   1. A standard consisting of either holmium oxide glass or a solution of holmium oxide in sulfuric or perchloric acid shall be used to verify the operation of the UV/VIS instrument.
B. For each instrument, the laboratory must establish wavelength specifications for the holmium oxide standard and include acceptable wavelength tolerances. The tolerances may be derived from instrument manufacturer’s specifications, the specifications of the standard, and/or repeated instrument measurements.
C. Scan the standard following the established specifications and record the results. Compare the established peak wavelength specifications to the measured wavelengths and either mark the results on the spectrum or list them in tabular form.
D. If the values are not within acceptable limits and cannot be addressed through troubleshooting, remove the instrument from service until corrective measures can be taken. Corrections may include replacing the holmium oxide standard or performing maintenance.
E. A performance check is completed if the instrument is moved.
F. A performance verification is completed if a major repair is performed.
G. Maintain results in a logbook or a retrievable format.
4.2 Controls

A. A blank must be prepared and analyzed each day the instrument is used prior to analyzing evidentiary samples.

B. The blank must be absent of any significant absorption.

C. If the blank does not meet specified criteria, attempt to troubleshoot the issue. Corrections may include discarding unused solution or cleaning the cuvettes.

D. Maintain spectra in a logbook or a retrievable format.

4.3 Calibration

There are no calibration requirements.

5 Procedure

5.1 Spectrophotometer Operating Conditions

A. The wavelength range used for the UV/Vis analysis of most drug samples is 340 nm to 220 nm.

B. The range may be expanded to accommodate certain substances, such as alkyl nitrites and GHB.

5.2 Sample Preparation

1. Dissolve the sample in a solution appropriate for the substance.

2. Depending on the concentration of the sample, it may be necessary to dilute the solution.

3. Plant materials will require extraction. Mixtures and other substances may require extraction prior to analysis.

5.3 Sample Analysis

1. Collect a spectrum of the sample in the appropriate solution.

2. A “pH shift” may be performed on samples in acidic solutions by adding concentrated sodium hydroxide until the solution is basic.

3. Print each spectrum and label with laboratory case number, exhibit number (where applicable), date, examiner’s handwritten initials and method of sample preparation (if not listed on the worksheet), and retain in the case record.

4. Retain instrument operating parameters in the case record or in a retrievable format.

5.4 Maintenance

A. Perform maintenance as needed.

B. Record all maintenance on the Equipment Log (LAB-405).

6 Interpretation

1. Evaluate the sample’s UV spectrum by comparing it to reference literature or to one or more UV spectra derived from known samples.

2. Document the test and results in the case record.

   a) A checkmark (√) may be used to indicate that the test was performed.
b) A plus sign (+) may be used to indicate that the sample spectrum supports the identification of the reported substance or substance class.

c) If the sample spectrum is interpreted as a match, document the source of any reference spectrum to which the sample spectrum is being compared. Use the approved list of reference abbreviations if applicable; if some other reference is used, provide a complete citation.

d) The interpretation of a spectrum may be documented directly on the spectrum itself.

7 Limitations

A. An ultraviolet spectrum is not specific, and a positive identification cannot be made exclusively on the basis of UV/VIS analysis.

B. Not all substances absorb ultraviolet light; therefore the lack of absorbance or a flat-line spectrum is not necessarily an indication that a sample contains no controlled substances.

C. The absorbance of a substance at any given wavelength may be modified by the presence of other compounds that also absorb at that wavelength. Additional sample preparation may be required to remove interfering compounds.

8 Advantages

A. The test is quick and easy to perform. It may provide a quick and easy quantitation of some drugs/diluents.

B. There is typically very little sample preparation required.

C. UV/Vis analysis is a useful screening tool and may provide information regarding the general concentration of the sample (strong, average or weak) and the presence or absence of some diluents and adulterants.

D. This is usually a non-destructive technique and the sample can be recovered for other testing procedures, if necessary.

9 Literature References and Supporting Documentation


SD-06-02 FOURIER TRANSFORM INFRARED (FTIR) SPECTROPHOTOMETRY

1 Scope
A non-destructive analytical technique used for the characterization and identification of seized drug evidence. Infrared spectrophotometers are considered significant equipment for the Seized Drug discipline.

2 Safety
A. Use appropriate safety equipment when preparing reagents.
B. Refer to the SDS for additional safety information for specific chemicals.

3 Equipment and Materials
- Fourier Transform Infrared Spectrophotometer
- Attenuated Total Reflectance (ATR) attachment
- Agate mortar and pestle
- Hydraulic press and KBr die or hand press
- Potassium bromide (KBr), dry
- NaCl or KBr windows (e.g., 2mm x 13 mm)
- Nujol
- Laboratory oven
- Special cells for liquids or vapors

4 Standards, Controls, and Calibration

4.1 Performance Verification/Check
A. Conduct a performance check on the instrument at least once each quarter of the calendar year using a polystyrene standard.
B. Scan the polystyrene film and display the results in percent transmittance versus the wavenumber.
   1. Examine the resulting spectrum for peaks at 3060, 1601, 1583, and 1028 cm⁻¹.
   2. Values must be within 4 cm⁻¹ of the expected values to pass.
C. If the values are not within acceptable limits and cannot be addressed through troubleshooting, remove the instrument from service until corrective measures can be taken. Corrections may include replacing the polystyrene standard or performing maintenance.
D. A performance check is completed if the instrument is moved.
E. A performance verification is completed if a major repair is performed.
F. Maintain spectra in a logbook or a retrievable format.

4.2 Controls
A. For the ATR, a blank will be performed each day the instrument is used prior to analyzing evidentiary samples.
B. For salt pellets and salt cells, a salt blank will be collected at least once each quarter when in use.

C. The blank must be absent of any significant transmittance of controlled substances or dangerous drugs.

D. If the blank does not meet specified criteria, attempt to troubleshoot the issue. This may include cleaning of the cell or accessory and/or discarding unused salt.

E. Maintain spectra in a logbook or a retrievable format.

4.3 Calibration

No calibration requirements.

5 Procedure

5.1 Sample Preparation

1. Use appropriate extraction and clean-up procedures as necessary to isolate the sample. This may require the conversion of the sample to a suitable salt form prior to analysis.

2. Methods of introducing the sample into the instrument for analysis include the following:

a) **Liquid samples can be analyzed as a thin film between two NaCl or KBr (salt) cells.**

b) **Volatile can be scanned using a special vapor phase cell.**

c) **Solid samples can be milled with dry KBr, KCl, NaCl, or a similar matrix to produce a fine powder. The powder is pressed into a thin pellet using a die and a hydraulic or hand press.**

d) **For cast film solid samples, dissolve a small amount of sample in a suitable solvent and place the solution on a single NaCl or KBr cell. Evaporate the solvent and scan the thin film remaining.**

e) **For smeared solid samples, mix a small amount of the powdered substance with a drop of Nujol to form a mull and smear it on a NaCl or KBr cell.**

f) **No sample preparation is typically needed when using the ATR attachment.**

5.2 Sample Analysis

1. Collect and print spectra with a resolution of at least 4 cm\(^{-1}\) from 4000 cm\(^{-1}\) to at least 600 cm\(^{-1}\) versus percent (%) transmittance. Spectral peaks should be of sufficient intensity to make an accurate comparison to known reference standards or published spectral data.

2. Label spectra with laboratory case number, exhibit number, date, examiner’s handwritten initials, and method of sample preparation (if not shown on the worksheet) and retain in the case record.

3. Retain instrument operating parameters in the case record or in a retrievable format.

4. Document the confirmation of the unknown spectra to a known reference and indicate the source of the reference in the case record (published or otherwise lab generated).
5.3 Maintenance

1. Perform maintenance as needed.

2. Record all maintenance on the Equipment Log (LAB-405).

6 Interpretation

A. For confirmation, the unknown spectrum must match spectra from known standards or libraries. A match is made when the following information compares favorably:

1. Overall appearance of the spectrum

2. Presence of absorption bands unique to that compound

B. The approved library reference list will be used to indicate the source of the reference.

7 Limitations

A. The sample must be relatively pure for positive identification.

B. For an accurate comparison of an unknown spectrum to a standard spectrum, both samples (the sample and reference) must be in the same salt form. Some compounds may produce different crystal structures that can result in slightly different infrared spectra.

C. FTIR analysis cannot usually be used to distinguish optical isomers.

8 Advantages

A. FTIR analysis is specific for the identification of controlled substances, dangerous drugs, and diluents and can be used as a confirmatory test.

B. FTIR analysis is normally not a destructive test and the sample can be recovered for additional testing procedures, if necessary.

C. The infrared spectrum of the majority of controlled substances and other substances routinely identified is specific to that compound and may be used for identification.

D. An unknown infrared spectrum can be quickly compared to known compounds found in drug libraries stored in the computer and then confirmed using published data from a reliable source or in-house spectra produced from known standards.

9 Literature References and Supporting Documentation


SD-06-03 GAS CHROMATOGRAPHY / MASS SPECTROMETRY (GC/MS)

1 Scope
An analytical technique for the characterization and identification of seized drug evidence. Gas chromatographs with mass spectroscopy detectors are considered significant equipment for the Seized Drug discipline.

2 Safety
A. Use appropriate safety equipment when preparing reagents and handling volatile chemicals. Refer to the SDS for additional safety information for specific chemicals.
B. Use properly secured high-pressure gas cylinders.
C. Use caution around hot surfaces such as oven interiors and injection and detector ports.

3 Equipment and Materials
- Gas chromatograph/mass spectrometer (GC/MS)
- Helium
- Auto-sampler vials and caps (where applicable)
- Microliter syringe (where applicable)

4 Standards, Controls, and Calibration
4.1 Performance Verification/Check
A. Conduct a performance check on the GC/MS at least monthly.
   1. A reference standard or reference standard mixture will be analyzed. Additional standards may be prepared and analyzed as needed or as defined in a local policy.
   2. The peak(s) of interest must be resolved and the mass spectra obtained must match a known reference. Maintain the resulting chromatograms in a logbook or a retrievable format.
   3. If the reference standard or reference standard mixture do not meet specified criteria and cannot be addressed through troubleshooting, remove the instrument from service until corrective measures can be taken. Corrections may include preparing a new standard or performing maintenance on the GC/MS.
B. The mass spectrometer will be tuned using a standard of PFTBA to ensure that the mass-to-charge ratios (m/z) are assigned correctly and to detect leaks.
   1. The laboratory must establish specifications for a successful tune. The tune criteria may be derived from instrument manufacturer's specifications and/or repeated instrument measurements.
      a) Laboratories must use the tune criteria for the instrumental analysis of Cannabis sativa L. as defined in this manual.
   2. If the tune does not meet specified criteria and cannot be addressed through troubleshooting, remove the instrument from service until corrective measures can be taken. Corrections may include performing maintenance.
C. A performance check is completed if the instrument is moved.
D. A performance verification is completed if a major repair is performed.
E. Records of all tunes (meeting and not meeting criteria), as well as corrections taken, will be maintained in a logbook or a retrievable format.
4.2 Controls

A. A solvent blank must be prepared and analyzed each day the instrument is used prior to analyzing samples. Additional blanks may be prepared and analyzed as needed or as defined in local policy. Maintain the resulting chromatograms in a logbook or a retrievable format.

B. A method blank is required when there is an insufficient amount of material for the analysis of two independent samples. The method blank must be prepared using the same parameters as the evidence sample. Maintain the resulting chromatograms in the case record or in a retrievable form.

C. The blank(s) must be absent of controlled substances or dangerous drugs.

D. If the blank(s) do not meet specified criteria, attempt to troubleshoot the issue. Corrections may include discarding of any unused solvent.

4.3 Calibration

No calibration requirements.

5 Procedure

5.1 GC/MS Operating Conditions

1. Use appropriate temperature programs and adjust other critical parameters to ensure that the suspected substance(s) will elute during data collection. The program should allow for a reasonable time for unknown or unexpected compounds to elute.

2. The method must use the current successful tune file.

3. Print and retain the program parameters in the case record or in a retrievable format.

5.2 Sample Preparation and Analysis

1. Extract samples into a suitable solvent before injecting into the instrument.

2. Analyze sample extracts and other controls, blanks, and/or standards, as appropriate.

3. Evaluate the GC/MS total ion chromatogram (TIC) and spectra of reported substances and other compounds of interest. Document the following:
   a) Complete Total Ion Chromatogram (TIC) of the sample
   b) Each sample mass spectrum that is used to confirm the identification of a reported substance
   c) Mass spectra of compounds of interest, as determined by the analyst
   d) Document observations of peaks that were evaluated on the primary TIC

4. Analyst discretion will be used when selecting peaks for observation based on analytical scheme and circumstances of the case. Samples which contain multiple controlled substances require the identification of the substance with the highest penalty.
   a) For peaks corresponding to a controlled substance, print the mass spectra and indicate the substance name on the peak or with another indicator
b) For peaks corresponding to a non-controlled substance matching a known reference, print the mass spectra and/or indicate the substance name on the peak or with another indicator.

c) For peaks not corresponding to any known reference, print the mass spectra and/or indicate “unidentified”, “unknown” or another indicator on the peak.

d) Peaks not evaluated are not marked.

5. Label each printout with:

a) Laboratory case number

b) Exhibit number (where applicable)

c) Date

d) Examiner’s handwritten initials (or secure electronic equivalent)

e) The method of sample preparation, if not documented on the worksheet

6. Document the comparison of the spectrum of each reported substance to a known reference spectrum and indicate the source of the reference. If the reference used for comparison has multiple entries in its library for the substance being identified, a unique identifier to the reference spectrum must be documented.

5.3 Retention Time Analysis (if applicable)

1. Select the appropriate reference standard for comparison with the unknown substance.

2. Analyze the prepared reference standard and the second prepared unknown sample(s) and compare the retention time of the peaks. The standard retention time will be valid until:

a) The method has changed, or

b) The column has been trimmed or replaced.

3. Print and retain the chromatograms of all relevant samples, blanks, and standards in the case record. These chromatograms will be labeled with

a) Laboratory case number

b) Corresponding exhibit number (where applicable)

c) Date

d) Examiner’s initials,

e) Method of sample preparation (if not shown on the worksheet)

f) Standard chromatograms must also contain a traceable lot number of the standard.

4. The percent error between retention times of the reference standard and unknown sample must be one percent or less and the calculation shall be documented in the case record.

\[
Percent\ Error = \frac{|\text{retention}_{\text{std}} - \text{retention}_{\text{unk}}|}{\text{retention}_{\text{std}}} \times 100
\]

Note: If the calculation is performed by a validated instrumental macro or software, the calculation is not required to be documented in the case record.
5.4 Maintenance
   1. Each laboratory must have a scheduled maintenance plan.
   2. Record all maintenance on the Equipment Log (LAB-405).

6 Interpretation
   A. For confirmation, the unknown spectrum must be compared to known standards or libraries. A match is made when the following information compares favorably:
      1. Overall appearance of the spectrum
      2. Presence of the ions unique to that compound
   B. The approved library reference list will be used to indicate the source of the reference.

7 Limitations
   A. When analysis by GC/MS is unable to provide positive identification, another technique (FTIR, derivatization, etc.) may be utilized to provide positive identification. For example, certain stereo- and geometric isomers produce identical or very similar results.
   B. Some compounds may not be suitable for GC/MS analysis due to a variety of factors. For example, high injection port temperatures cause some compounds to break down before they are ionized, preventing their identification.
   C. It may be difficult to identify individual compounds in a homologous series.

8 Advantages
   A. Generally, mass spectra of controlled substances are specific to single compounds and may be used for identification.
   B. It may be possible to separate and identify complex mixtures that are difficult to separate through ordinary clean-up procedures.
   C. The technique is useful for analyzing small sample amounts that may be difficult to identify using other techniques.
   D. An autosampler, which increases the efficiency of analysis of numerous samples and functions unattended, may be attached to the GC/MS.
   E. The technique can be enhanced when extracts are analyzed two or more times using columns with differing stationary phases.

9 Literature References and Supporting Documentation
SD-06-04 GAS CHROMATOGRAPHY / FLAME IONIZATION DETECTION (GC/FID)

1 Scope
An analytical technique for the retention time analysis of seized drug evidence. Gas chromatographs with flame ionizing detectors are significant equipment for the seized drug discipline.

2 Related Chapters
Quantitation by Gas Chromatography with Internal Standard (SD-07-01)

3 Safety
A. Use appropriate safety equipment when preparing reagents and handling volatile chemicals. Refer to the SDS for additional safety information for specific chemicals.
B. Use properly secured high-pressure gas cylinders.
C. Use caution around hot surfaces.

4 Equipment and Materials
- Gas Chromatograph (GC) equipped with a Flame Ionization Detector (FID)
- Appropriate carrier and fuel gases
- Auto-sampler vials and caps (where applicable)
- Microliter syringe (where applicable)

5 Standards, Controls, and Calibration
5.1 Performance Verification/Check
A. A reference standard or standard mixture must be analyzed each day the instrument is used prior to analyzing samples. Additional standards may be prepared and analyzed as needed or as defined by local policy.
B. The peak(s) of interest must be resolved.
C. If the peak(s) of interest is not resolved and cannot be addressed through troubleshooting, remove the instrument from service until corrective measures can be taken. This can include performing maintenance or preparing a new standard or standard mixture.
D. Records will be maintained in a logbook or a retrievable format.

5.2 Controls
A. A solvent blank must be prepared and analyzed each day the instrument is used prior to analyzing evidentiary samples.
B. A method blank is required when there is an insufficient amount of material for the analysis of two independent samples. The method blank must be prepared using the same parameters as the evidentiary sample.
C. The blank(s) must be absent of controlled substances or dangerous drugs.
D. If the blank(s) does not meet specified criteria, attempt to troubleshoot the issue. Corrections may include discarding any unused solvent.
E. Records will be maintained in a logbook or a retrievable format.
5.3 Calibration

No calibration requirements.

6 Procedure

6.1 Retention Time Analysis

1. Select the appropriate reference standard for comparison with the unknown substance. Ensure that the compounds are in the same chemical form (i.e. both in the base or salt form).

2. Analyze the reference standard and the unknown sample(s) and compare the retention time of the peaks. The reference standard retention time will be valid until:
   a) The method has changed, or
   b) The column has been trimmed or replaced.

3. Print and retain the chromatograms of all relevant samples, blanks, and reference standards in the case record. These chromatograms will be labeled with:
   a) Laboratory case number
   b) Corresponding exhibit number (where applicable)
   c) Date
   d) Examiner’s initials
   e) The method of sample preparation, if not documented on the worksheet

4. Drug reference standard chromatograms must also contain a traceable lot number.

5. Instrument operating parameters will be retained in the case record or in a retrievable format.

6.2 Maintenance

1. Each laboratory must have a scheduled maintenance plan.

2. Complete a performance check if the instrument is moved.

3. Complete a performance verification if a major repair is performed.

4. Record all maintenance on the Equipment Log (LAB-405).

7 Interpretation

A. Examine the resulting chromatogram(s) to determine if the separation between components is adequate for the intended purpose.

B. The percent error between retention times of the standard and unknown samples must be one percent or less.

\[
\text{Percent Error} = \frac{|\text{retention}_{\text{std}} - \text{retention}_{\text{unk}}|}{\text{retention}_{\text{std}}} \times 100
\]
8 Limitations
   A. Two or more compounds, especially those similar in chemical structure, can have the same retention time under identical GC conditions.
   B. Co-eluting compounds may mask the peak of interest.
   C. An elevated injection port temperature can result in partial decomposition of certain compounds.
   D. Concentration may affect retention time.

9 Advantages
   A. Excellent technique for separating chemical components and assists in the identification of complex mixtures.
   B. Relatively simple sample preparation.
   C. Technique can be enhanced when extracts are analyzed two or more times using columns with differing stationary phases.
   D. GC conditions, such as the carrier gas flow rate and the oven temperature program, can be adjusted to allow for greater separation of analytes.

10 Literature References and Supporting Documentation


SD-06-05 HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

1 Scope
An analytical technique for the characterization and identification of seized drug evidence. Liquid chromatographs are significant equipment for the Seized Drugs discipline.

2 Safety
A. Use appropriate safety equipment such as eye protection, gloves, and lab coat, when preparing reagents and handling volatile chemicals. Refer to the SDS for additional safety information for specific chemicals.

B. Elution solvents should be prepared in a well ventilated area, preferably a hood, especially when solvents such as acetonitrile are used. Waste solvents should be disposed of in a proper manner.

3 Equipment and Materials
- High performance liquid chromatograph system including:
  - solvent degasser
  - pump system
  - injector
  - detector such as photodiode array detector
- Microliter syringe (with special tip to match injector valve)
- Equipment to prepare elution solvents such as:
  - balances
  - graduated cylinders
  - volumetric flasks
  - pipets
  - pH meter
  - magnetic stirrer
- Syringe filter for sample filtration
- High purity solvents (HPLC grade or better) such as:
  - water
  - methanol
  - acetonitrile

4 Standards, Controls, and Calibration
4.1 Performance Verification/Check
A. LC
   1. Conduct a performance check at least once per month when in use.
      a) A reference standard or reference standard mixture shall be used to verify the condition of the instrument.
      b) The peak(s) of interest must be resolved.
      c) If the peak(s) of interest is not resolved and cannot be addressed through troubleshooting, remove the instrument from service until corrective measures can be taken.
2. A performance check is completed if the instrument is moved.
3. A performance verification is completed if a major repair is performed.
4. Maintain results in a logbook or a retrievable format.

B. UV
1. Conduct performance check on the UV/VIS detector at least once each quarter of the calendar year.
2. A holmium oxide standard or other standard recommended by the manufacturer shall be used to verify the condition of the UV/VIS detector.
3. The laboratory must establish wavelength specifications for the standard and include acceptable wavelength tolerances. The tolerances may be derived from manufacturer’s specifications, the specifications of the standard, and/or repeated measurements.
4. If the values are not within acceptable limits, remove the instrument from service until corrective measures can be taken and the instrument is placed back into service.
5. Verify performance if the instrument is moved or if a major repair is performed.
6. Maintain results in a logbook or a retrievable format.

4.2 Controls
A. A solvent blank must be prepared and analyzed each day the instrument is used for evidentiary samples. Additional blanks may be prepared and analyzed as needed or as defined in a local policy.
B. A method blank will be required when there is an insufficient amount of material for the analysis of two independent samples. The method blank must be prepared using the same parameters as the evidence sample. Maintain the resulting chromatograms in the case record or a retrievable format.
C. The blank(s) must be absent of controlled substances or dangerous drugs.
D. If the blank(s) does not meet specified criteria, attempt to troubleshoot the issue. Corrections may include discarding any unused solvent.
E. Maintain the resulting chromatograms and spectra in a logbook or a retrievable format.

4.3 Calibration
No calibration requirements

5 Procedure
5.1 Analysis
1. Select the appropriate drug reference standard for comparison with the unknown substance.
   a) Prepare the drug reference standard and the evidence sample using the same solvent and/or extraction procedure.
   b) When possible, prepare solutions for injection in the elution solvent.
2. Verify the appropriate program parameters for the HPLC.
3. Analyze the prepared drug reference standard and prepared unknown sample(s) and compare the retention time and UV absorption spectra of the peaks.

4. Print and retain the chromatograms and spectra of all relevant samples and standards in the case record. The chromatograms and spectra will be labeled with:
   a) Laboratory case number
   b) Exhibit number (where applicable)
   c) Date
   d) Examiner handwritten initials
   e) The method of sample preparation, if not documented on the worksheet
   f) Instrument operating parameters, including the column type and mobile phase, will be retained in the case record or in a retrievable format.

5.2 Maintenance
   1. Each laboratory must have a scheduled maintenance plan.
   2. Record all maintenance on the Equipment Log (LAB-405).

6 Interpretation
   A. Evaluate the UV spectrum of the sample by comparing it to reference literature or to one or more UV spectra derived from known samples.
   B. The percent error between retention times of the standard and unknown samples must be one percent or less, if applicable.

\[
\text{Percent Error} = \frac{|\text{retention}_{\text{std}} - \text{retention}_{\text{unk}}|}{\text{retention}_{\text{std}}} \times 100
\]

7 Limitations
   A. Two or more compounds, especially those similar in chemical structure, can have similar retention times and UV absorption spectra under the same chromatographic conditions.
   B. Certain solvents can cause decomposition of some samples, such as the hydrolysis of heroin.
   C. HPLC retention times and UV absorbance spectra are a category B test.
   D. Not all substances absorb UV or visible light, and therefore, these compounds would not be detected with a photodiode array detector.
   E. The absorbance of many compounds depends on the solvent used, which can make comparison to standards difficult.

8 Advantages
   A. Excellent technique for separating chemical components
   B. Allows quantitation of complex mixtures
   C. Useful technique for separating analytes that are thermally unstable or not readily volatile
   D. Relatively simple sample preparation
   E. Separation can be enhanced by varying the column or solvent system.
F. Sensitivity and linear detection range can be increased by monitoring various or specific wavelengths.

G. Can be nondestructive; fractions can be collected for further analysis

9 Literature References and Supporting Documentation


1 Scope
An analytical technique for the characterization and identification of seized drug evidence. Liquid chromatographs with mass spectroscopy detectors are considered significant equipment for the Seized Drugs discipline.

2 Safety
A. Use appropriate safety equipment such as eye protection, gloves, and lab coat, when preparing reagents and handling volatile chemicals. Refer to the SDS for additional safety information for specific chemicals.

B. Elution solvents should be prepared in a well ventilated area, preferably a hood, especially when solvents such as acetonitrile are used. Waste solvents should be disposed of in a proper manner.

3 Equipment and Materials
- Ultra performance liquid chromatograph system including:
  - Solvent degasser
  - Pump system
  - Injector
  - Detectors such as photodiode array detector (pda) and single quadrupole mass detector
- Microliter syringe (where applicable)
- Autosampler vials and caps with pre-slit silicone septa
- Equipment to prepare elution solvents such as:
  - Balances
  - Graduated cylinders
  - Volumetric flasks
  - Pipettes
- Particulate filters for sample filtration
- High purity solvents (LC grade or better) such as:
  - Water
  - Methanol
  - Acetonitrile
  - Isopropyl alcohol
- Nitrogen
4 Standards, Controls, and Calibration

4.1 Performance Verification/Checks using Standards
   A. Conduct a performance check of the PDA and mass detector monthly or more often, as needed, using a reference standard. The performance check must meet specified criteria for the instrument to be in service.
   B. The UPLC/MS operational status will be checked by running a blank before the reference standard.
   C. For each UPLC/MS instrument, the laboratory must establish specifications for the reference standard to pass a performance check. Criteria may be derived from instrument manufacturer’s specifications and/or repeated instrument measurements.
   D. If the blank does not meet specified criteria, attempt to troubleshoot the issue. Corrections may include preparing a new blank, mobile phase and/or performing maintenance. If troubleshooting/appropriate corrections are unsuccessful, remove the instrument from service until corrective measures can be taken and the instrument is returned to service.
   E. Complete a performance check if the instrument is moved.
   F. Complete a performance verification if a major repair is performed on the instrument.
   G. Instrument records will be maintained in a logbook or retrievable format.

4.2 Controls
   A. A solvent blank must be analyzed each day the instrument is used for evidentiary samples. Additional blanks may be prepared and analyzed as needed or as defined in a local policy.
   B. A method blank will be required when there is an insufficient amount of material to analyze two independent samples. The method blank must be prepared using the same parameters as the evidentiary sample. Maintain the resulting chromatograms in the case record or a retrievable format.
   C. The daily blank must be absent of controlled substances or dangerous drugs.
   D. If the daily blank does not meet specified criteria, attempt to troubleshoot the issue. Corrections may include discarding any unused solvent.
   E. Maintain the resulting chromatograms and spectra in a logbook or a retrievable format.

5 Procedure

5.1 UPLC/MS Operating Conditions
   1. Use appropriate and approved methods to ensure that the suspected substance(s) will elute during data collection.
   2. Retain method parameters in the case record or in a retrievable format.

5.2 Sample Preparation for Preliminary Analysis
   1. Extract samples into the appropriate solvent.
   2. All samples and blanks must be run through a particulate filter before analysis.
   3. Select the appropriate screening method for the UPLC.
   4. Analyze sample extracts, blanks, and/or standards as appropriate.
5. Evaluate the UV spectra, molecular weight ion, and/or retention time.

6. Print and retain the chromatograms and spectra of all relevant samples and standards in the case record.

7. Label each print out with
   a) Laboratory case number
   b) Exhibit number (where applicable)
   c) Date
   d) Examiner's handwritten initials or electronic equivalent
   e) Method of sample preparation (if not shown on the worksheet)

8. Instrument operating parameters, including the column type and mobile phase will be retained in the case record or in a retrievable format.

5.3 Maintenance
1. Each laboratory must have a scheduled maintenance plan.
2. Record all maintenance on the Equipment Log (LAB-405).

6 Interpretation
A. Evaluate the UV spectrum of the sample by comparing it to a laboratory generated reference library or to a reference standard(s). If the spectra are similar, this supports the identification of the reported substance or substance class.

B. Evaluate the mass spectrum of the sample by comparing it to a laboratory generated reference library or to a reference standard(s).
   1. Result will be molecular weight +1 amu in positive mode or -1 amu in negative mode
   2. For preliminary analysis, if the molecular weight is +/- 1 amu when compared to a laboratory generated or published reference, it is considered to be an affirmative result.

C. The percent error between retention times of the known and unknown samples must be 1% or less to be considered to be an affirmative test. The calculation must be documented in the case record.

\[
\text{Percent Error} = \left| \frac{\text{retention}_{std} - \text{retention}_{unk}}{\text{retention}_{std}} \right| \times 100
\]

D. For exam counting, the total number of injections will be recorded in LIMS. Observations may be recorded on the spectra or on the worksheet.

7 Limitations
A. Two or more compounds, especially those with a similar chemical structure, can have similar retention times, UV absorption spectrum, and molecular weights under the same chromatographic conditions.

B. Certain solvents can cause decomposition of certain samples.

C. LC retention times, UV absorbance spectra, and molecular weight only spectrometry are not considered confirmatory tests.
D. Not all substances absorb UV or visible light and therefore, these compounds would not be detected with a photodiode array detector.

E. The absorbance of many compounds depends on the solvent used which can make comparison to standards difficult.

8 Advantages
A. Excellent technique for separating chemical compounds.
B. Useful technique for analyzing compounds which are thermally unstable or not readily volatile.
C. Adjusting the voltage in the mass detector can optimize insource fragmentation.
D. Relatively simple sample preparation.
E. Separation can be enhanced by varying the column or solvent system.
F. Sensitivity and linear detection range can be increased by monitoring various or specific wavelengths.

9 Literature References and Supporting Documentation


Printed copy is uncontrolled. Refer to electronic copy for current version.
SD-06-07  BALANCES AND SCALES

1 Scope
To provide instructions for the use and maintenance of balances and scales in the laboratory. Balances are considered significant equipment for the Seized Drugs discipline.

2 Related Chapters/Documents
Examination of Seized Drug Evidence (SD-03-01)
CLS Manual: Laboratory Equipment
CLS Manual: Standards, Reference Materials/Collections, Databases, and Controls

3 Safety
Use proper lifting techniques and caution when handling heavy items.

4 Equipment and Materials
• Analytical balance
• Top loading balance
• Bulk balance or scale
• Standard weights
• Weighing vessel (weighing boats, weighing paper, etc.)
• Spatula

5 Standards, Controls, and Calibration
5.1 Performance Verification/Check
A. Balance accuracy will be checked using standard weights at least once each quarter of the calendar year.
B. Conduct a performance check whenever a balance or scale is moved from one location to another.
C. The values at which the balances and scales must be assessed include (where the balance is capable):
   1. Analytical balances: 10mg, 100mg, 1g, 10g
   2. Top Loader balances: 1g, 4g, 10g, 100g, 200g, 400g
   3. Bulk Scales: 10lbs, 25lbs, 50lbs
D. Each laboratory will establish tolerances specific to their balances for the masses checked. The tolerances may be derived from balance manufacturer specifications, repeated measurements and/or calibration certificates.
E. Balances that are out of tolerance and cannot be corrected through troubleshooting, will be removed from service until appropriate corrections can be made.
F. A performance check is completed if the instrument is moved.
G. A performance verification is completed if a major repair is performed.
H. A logbook with the performance verification/check results will be maintained.
5.2 Calibration
   A. Balances must be calibrated annually by an approved vendor.
   B. Standard weights must be calibrated every 3 years by an approved vendor. (*CLS Manual – Laboratory Equipment*)

6 Procedures

6.1 Instructions
See “Procedure for Weighing Samples” within *Examination of Seized Drugs Evidence (SD-03-01)*.

6.2 Maintenance
   1. Keep balances clean and leveled at all times.
   2. Record all maintenance on the Equipment Log (LAB-405).

7 Precautions
   A. The appropriate balance will be used for the weight being measured and the level of precision required.
   B. Care should be taken to not overload a balance. Exceeding the capacity of the balance can damage the equipment.
   C. Balances equipped with a level indicator should be kept level for proper function.

8 Records
Performance check/verification records (LAB-405/LAB-408)
Maintenance or repair records (LAB-405)

9 Literature References and Supporting Documentation
Manufacturer’s equipment manual(s)
SD-06-08 MECHANICAL PIPETTES

1 Scope
To provide instructions for the use and maintenance of mechanical pipettes in the laboratory. Pipettes are considered significant equipment for the Seized Drugs discipline.

2 Related Chapters/Documents
Instrumental Analysis of Cannabis sativa L. (SD-03-06)
CLS Manual: Laboratory Equipment
CLS Manual: Standards, Reference Materials/Collections, Databases, and Controls

3 Safety
A. Use appropriate safety equipment when handling chemicals.
B. Refer to the SDS for additional safety information for specific chemicals.

4 Equipment and Materials
- Eppendorf Reference 2 pipette
- Eppendorf Repeater M4 pipette

5 Standards, Controls, and Calibration
5.1 Calibration
A. Pipettes must be calibrated annually by an approved vendor.
B. Complete a performance verification and document on the LAB-408 when the pipette is returned from the calibration vendor prior to being placed back into service.

5.2 Performance Verification/Check
A. Conduct a performance check approximately every 6 months after the calibration performance verification or whenever maintenance is performed.
B. A performance verification or performance check will be completed using a gravimetric method to collect four or more data points at three different volumes across the range of the pipette to verify accuracy of the delivery volumes.
   1. The coefficient of variation (%CV) must be within 1%.
   2. The accuracy must be within the following range:
      a) For the Reference 2 20-200 µL pipette: ± 2.5%
      b) For the Reference 2 100-1000 µL pipette: ± 3%
      c) For the Repeater M4 pipette: ± 1%
C. The data collected from the gravimetric study will be documented.
D. The documentation will be maintained in a logbook or retrievable format.
C. Results of the performance check (pass or fail) will be recorded and maintained on the LAB-405.
6 Instructions

6.1 Eppendorf Reference 2 Pipette Instructions

1. Rotate the control button to set the desired volume.
2. Attach the appropriate pipette tip to the end of the pipette.
3. Aspirating liquid
   a) Press down on the control button until the first stop.
   b) Immerse the pipette tip vertically in the liquid.
   c) Maintain the immersion depth and let the control button slide back slowly.
   d) Wait approximately 3 seconds and then remove the pipette tip from the liquid.
4. Dispensing liquid
   a) Place the pipette tip on the inner wall of the container at a slight angle.
   b) Slowly press down the control button to the first stop.
   c) Wait until the flow of liquid stops and then press down the control button to the second stop to completely empty the pipette tip.
   d) Hold down the control button and wipe the pipette tip on the inner wall of the container.
5. Eject the pipette tip by pressing the control button down all the way.

6.2 Eppendorf Repeater M4 Pipette Instructions

1. Attach the appropriate dispensing tip to the end of the pipette.
2. Select the desired dispensing volume using the selection dial.
3. To aspirate liquid, immerse the pipette into the liquid and slowly and steadily slide the filling lever up.
4. Press the operating lever to trigger the reverse stroke and dispense the reverse stroke into a waste container. This will stop the display from blinking and the steps display will be set to 0.
5. To dispense liquid, push the operating lever down as far as it will go. The display will show the number of dispensing steps performed. Then let the operating lever slide back to its initial position to complete the step.
6. Push the operating lever down again to perform the next dispensing step.
7. Repeat until all desired dispensing steps are completed and/or the pipette needs to be refilled to continue dispensing.
8. If liquid still remains in the pipette and all dispensing steps have been completed, empty the dispensing tip by pushing the filling lever down as far as it will go.
9. Push the operating lever all the way down to eject the dispensing tip.

7 Records

Equipment Log (LAB-405)
8 Literature References and Supporting Documentation

07 QUANTITATIVE ANALYSIS

SD-07-01 QUANTITATION BY GAS CHROMATOGRAPHY WITH INTERNAL STANDARD

1 Scope
To establish a procedure to determine the concentration of a seized drug evidence sample using gas chromatography with an internal standard

2 Safety
A. Use appropriate safety equipment when preparing reagents and handling volatile chemicals. Refer to the SDS for additional safety information for specific chemicals.
B. Use properly secured high-pressure gas cylinders.
C. Use caution around hot surfaces.

3 Related Chapters
Standards and References (SD-02-01)
Reagents (SD-02-04)
Examination of Seized Drug Evidence (SD-03-01)
Instructions for Seized Drugs Worksheet (SD-03-02)
Gas Chromatography/Mass Spectrometry (SD-06-03)
Gas Chromatography/Flame Ionization Detection (SD-06-04)

4 Equipment and Materials
- Gas chromatograph
- Appropriate carrier and fuel gases
- Injection syringe
- Analytical balance
- Pipettor, Class A volumetric pipette or flask
- Chloroform (Chromatographic Grade or Ultra-high Purity)
- n-Tetradecane (C-14)
- Methamphetamine HCl standard
- 1 M Sodium Carbonate and other solution as needed
- Purchased standards (NIST Traceable) of known concentration, as they become available

5 Standards, Controls, and Calibration
5.1 Performance Verification/Check
A. Semi-annually (at least five months but less than eight months apart), each instrument shall have, at a minimum, a three-point linearity plot with a correlation number ($R^2$) greater than or equal to 0.995 for the standard curve using a common controlled substance (e.g., Methamphetamine).
B. If major instrument repairs (e.g., replacement of the detector) are performed the linearity will be re-confirmed as part of the performance verification.
C. The requirement for linearity verification only applies when the instrument is in use.

D. Instrument performance verification, checks, and controls will be completed as defined in SD-06-04 or SD-06-03.

5.2 Internal Standard Solutions

A. Prepare an internal standard stock solution by dissolving the hydrocarbon appropriate to the substance to be quantitated in a suitable solvent. Internal standards which may be used include n-C-14 for methamphetamine.

B. Final concentration of the internal standard in the quantitation samples should be approximately 1.0 mg per 1.0 mL chloroform or other suitable solvent.

C. If stock solution is stored for later use, it should be stored appropriately, such as in the refrigerator. It should be labeled with internal standard name, date of preparation, initials of analyst who prepared solution and final concentration of the internal standard. A reagent log must be kept for internal stock solutions.

D. The internal standard solution will be checked at the beginning of the sequence in order to demonstrate the absence of contaminants and to verify the internal standard.

5.3 Standards and Calibrators

A. Volumetric flasks

1. Borosilicate volumetric flasks must be calibrated every 10 years by an external vendor.

2. Soda-lime volumetric flasks must be calibrated every 5 years by an external vendor.

B. New calibrations will be performed using a prepared known standard and then verified using a second prepared known standard with a different lot number. If the result is not within 5% of expected result, the issue will be resolved prior to use on casework samples. Examples of resolutions may include re-injecting the standards or preparing new standards.

C. Saved calibrations will be verified using a prepared known standard (this standard cannot be the same used to previously calibrate instrument). If the result is not within 5% of the expected amount, the issue will be resolved prior to use on casework samples. Examples of resolutions may include re-injecting the standards or preparing new standards.

D. If saved calibration has not been used for 6 months, a new calibration will be performed.

6 Procedures

6.1 Preparation of Reference Standards

1. Weigh a known standard using the reference sample of the controlled substance to be quantitated. At least 5 mg of standard should be used. Document the amount and lot number of sample used.

2. Add sufficient or approximately 1.0 mL 1.0M Sodium Carbonate or other aqueous base to each of the standards.

3. Add an appropriate volume of internal standard solution to each of the standards by Class A volumetric pipette, a pipettor, or a Class A volumetric flask to give a solution with a concentration of about 1 mg/mL of the drug base. Mix thoroughly.

4. The reference standard may be capped and stored in a refrigerator for later use.

It will be labeled with
5. The calculated amount of the reference standard is determined by multiplying the weight of the reference standard used by the molecular weight of the base form, then dividing the result by the molecular weight of the salt form.

For example, the molecular weight of Methamphetamine base (149) divided by the molecular weight of Methamphetamine HCl (186) is 80%.

6.2 Preparation of Sample(s)

1. Obtain a representative sample from the item(s) being quantitated.
   a) If performing a composite quantitation, combine the representative samples and homogenize.
   b) If performing a quantitation on individual items, homogenize each item separately

2. Prepare two independent samples from the homogenization.

3. Weigh an appropriate amount of unknown sample directly into an appropriate container. Enough sample should be used to make the solution approximately 1 mg/mL.
   a) For example, 5mg into 5mL gives a concentration of 1mg/mL.
   b) Document the amount of sample used.

4. Add sufficient or approximately 1.0mL of 1.0M Sodium Carbonate or other aqueous base to sample.

5. Add an appropriate volume of internal standard solution to each sample by Class A volumetric pipette, a pipettor, or Class A volumetric flask to give a solution with a concentration of about 1 mg/mL of the drug base. Mix thoroughly.

6.3 Analysis

1. Retain instrument operating conditions in the case record or available in a retrievable format.

2. For a new calibration, inject a prepared known standard at least twice, average the resulting calibrating data, then verify the calibration using a second prepared known standard of a different lot number.

3. For a saved calibration, verify calibration using a prepared known standard (standard cannot be the same used to previously calibrate instrument).

4. When verifying a new or saved calibration, the result must be within 5% of the expected amount. Percent error calculated by:

\[
\text{%Error} = \left(\frac{\text{Observed} - \text{Expected}}{\text{Expected}}\right) \times 100
\]
5. If the results do not meet the expected amount, then the issue will be resolved prior to use on casework samples.

6. Analyze an aliquot of the internal standard solution at the start of each sequence in order to demonstrate the absence of contaminants in the solvent and to verify the internal standard.

7. Inject each prepared unknown sample once.

8. Analyze the prepared known standard again at the end of the sequence to verify the calibration. A NIST Traceable purchased standard may also be used for this check. If the result is not within 5% of the expected result, then the issue will be resolved prior to reporting results.

9. Print the chromatograms of all relevant samples and standards (including lot number) and retain in the case record. The chromatograms will be labeled with:
   a) Laboratory case number
   b) Exhibit number (where applicable)
   c) Date
   d) Examiner’s handwritten initials (or secure electronic equivalent)
   e) The method of sample preparation, if not documented on the worksheet

7 Interpretation

A. The concentration of the drug present is proportional to the ratio of the integrated area of the sample to the integrated area of the internal standard.

\[
\text{Concentration}_{\text{unknown}} = \frac{\frac{\text{Area}_{\text{Unknown}}}{\text{Area}_{\text{STD}}}}{\frac{\text{Area}_{\text{Standard}}}{\text{Area}_{\text{STD}}}} \times \frac{\text{Concentration}_{\text{Standard}} \times \text{Concentration}_{\text{Standard ISTD}}}{\text{Concentration}_{\text{Unknown ISTD}}}
\]

B. The concentration of the analyte will be calculated and reported in its salt form.

C. The reported value shall be the average of the results from the two independent samples and will be reported in accordance with guidelines in SD-01-01.

8 Limitations

A. Standards of known purity must be used.

B. The peak to be quantitated must be a single component peak and completely resolved.

9 Advantages

A. Samples containing complex mixtures can be quantitated.

B. Many different compounds can be analyzed on the same instrument and column by varying the GC conditions.

10 Records

Seized Drugs Worksheet (in LIMS or LAB-SD-01)
1 Scope
To establish a procedure to determine the concentration of methamphetamine HCl in a sample using High Performance Liquid Chromatography.

2 Safety
A. Use appropriate safety equipment such as eye protection, gloves, and lab coat, when preparing reagents and handling volatile chemicals. Refer to the SDS for additional safety information for specific chemicals.

B. Elution solvents should be prepared in a well ventilated area, preferably a hood, especially when solvents such as acetonitrile are used. Waste solvents should be disposed of in a proper manner.

3 Related Chapters
Standards and References (SD-02-01)
Reagents (SD-02-04)
Examination of Seized Drug Evidence (SD-03-01)
Instructions for Seized Drugs Worksheet (SD-03-02)
High Performance Liquid Chromatography (SD-06-05)

4 Equipment and Materials
- Refer to local documents for specific chemicals.
- HPLC
- Analytical balance
- Pipettor, Class A volumetric pipette or volumetric flask
- HPLC Grade Reagents
- Methamphetamine HCl reference standard
- Purchased standards (NIST Traceable) of known concentration, if available

5 Standards, Controls, and Calibration

5.1 Calibration Curve and Performance Verification/Check
A. Semi-annually (at least five months but less than eight months apart), each instrument shall have, at a minimum, a three-point linearity plot with a correlation number ($R^2$) greater than or equal to 0.995 for the standard curve methamphetamine HCl.

B. If major instrument repairs (e.g., replacement of the detector) are performed, the linearity will be re-confirmed as part of the performance verification.

C. The requirement for linearity verification only applies when the instrument is in use.

D. Instrument performance linearity verification and checks will be completed as defined in SD-06-05.
5.2 Standards and Calibrators

A. Volumetric flasks
   1. Borosilicate volumetric flasks must be calibrated every 10 years by an approved vendor.
   2. Soda-lime volumetric flasks must be calibrated every 5 years by an approved vendor.

B. HPLC
   1. New calibrations will be performed using prepared known standards.
   2. New and saved calibrations will be verified using one prepared known standard (this standard cannot be the same used to previously calibrate instrument). If the result is not within 5% of the expected result, the issue will be resolved prior to use of data generated on casework samples. Examples of resolutions may include re-injecting the standards or preparing new standards. Percent error is calculated by:

   \[
   \%\text{Error} = \frac{|\text{Observed} - \text{Expected}|}{\text{Expected}} \times 100
   \]

   3. If saved calibration has not been used for 6 months, a new calibration will be performed.

6 Procedures

Refer to local documents for additional instructions.

6.1 Preparation of reference standards

1. Document preparation of reference standards, including the lot numbers for traceability.
2. Weigh the reference standard samples into appropriate containers.
   a) At least 5 mg of standards should be used for each preparation.
   b) Place the samples into Class A volumetric flasks.
3. Prepare standards in HPLC grade water.
   a) Add an appropriate volume of water to the Class A volumetric flasks to produce the desired concentrations.
   b) Mix thoroughly and allow to rest.
4. The prepared reference standards may be capped and stored in a refrigerator for later use. They will be labeled with:
   a) Identity of the solution
   b) Lot number
   c) Date of preparation
   d) Initials of analyst who prepared solution
   e) Final concentration

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6.2 Preparation of Sample(s)
1. Obtain a representative sample from the item(s) being quantitated.
2. Prepare two independent samples from the representative sample.
3. Place samples in appropriate Class A volumetric flasks and dilute with HPLC grade water.
4. Mix thoroughly and allow to rest.

6.3 Analysis
1. Retain instrument operating conditions in the case record or in a retrievable format.
2. Run calibration samples per local instructions.
3. Analyze an aliquot of stock HPLC grade water used to prepare the unknown samples at the start of each sequence in order to demonstrate the absence of contaminants.
4. Analyze the prepared unknown samples.
5. Analyze the prepared standard again at the end of the sequence to verify the calibration. If the result is not within 5% of the expected result, then the issue will be resolved prior to reporting results.
6. Print the chromatograms of all relevant samples and standards (including lot number) and retain in the case record. These chromatograms will be labeled with:
   a) Laboratory case number
   b) Exhibit number (where applicable)
   c) Date
   d) Examiner’s handwritten initials (or electronic equivalent)
   e) The method of sample preparation, if not documented on the worksheet

7 Interpretation
A. The concentration of the analyte will be calculated and reported in its salt form.
B. The reported value will be the average of the results of the two independent samples and will be reported in accordance with guidelines in SD-01-01.

8 Limitations
A. Standards of known purity must be used.
B. The peak to be quantitated must be a single component peak and completely resolved.

9 Advantages
A. Samples containing complex mixtures can be quantitated.
B. Many different compounds can be analyzed on the same instrument and column by varying the LC conditions.

10 Records
Seized Drugs Worksheet (in LIMS or LAB-SD-01)
SD-07-03 QUANTITATION SAMPLING PROCEDURE

1 Scope
To establish a procedure to sample and prepare seized drugs exhibits for quantitation. Quantitation is only performed on confirmed methamphetamine exhibits for federal prosecution.

2 Related Chapters/Documents
Reporting Guidelines (SD-01-01)
Examination of Seized Drug Evidence (SD-03-01)
Instructions for Seized Drugs Worksheet (SD-03-02)
Quantitation by Gas Chromatography with Internal Standard (SD-07-01)
Quantitation by High Performance Liquid Chromatography (SD-07-02)
LIMS Manual: Seized Drugs Quantitation (LIMS-11-05)
CLS Manual: Laboratory Equipment
CLS Manual: Standards, Reference Materials/Collections, Databases, and Controls

3 Safety
A. Use caution when handling any unknown substance or chemical.
B. For possible hazardous materials, use appropriate personal protective equipment and fume hoods.
C. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

4 Equipment and Materials
- Weighing vessel (weighing boats, weighing paper, etc.)
- Spatula
- Mortar
- Pestle
- Butcher Paper
- Plastic Bags
- Top Loader Balance

5 Standards, Controls, and Calibration
A method blank including the mortar and pestle will be run as a negative control.

6 Procedure
6.1 Instructions for All Laboratories
A. Exhibits confirmed to contain fentanyl or a derivative will not be quantitated due to personnel safety risk.
B. Quantitation is not performed on liquids, tablets, or exhibits < 5 grams.
C. For exhibits greater than or equal to 5 grams, but less than 1 kilogram:
   1. If quantitation is requested and analysis has not been completed, the receiving laboratory will perform qualitative analysis, including an attempt to determine the salt form, and then forward the container with the item needing quantitation to the designated quantitation laboratory. (Questions regarding packaging may be resolved with the designated quantitation laboratory).
      a) Austin is the designated quantitation laboratory for Austin, Corpus Christi, El Paso, Houston, Laredo, Midland, Waco, and Weslaco.
      b) Garland is the designated quantitation laboratory for Abilene, Amarillo, Garland, Lubbock and Tyler.
      c) The designated quantitation laboratories may assist one another with quantitation work at their discretion.
   2. If quantitation is requested after analysis has been completed and the evidence has not been returned, the laboratory will forward the container with the item needing quantitation to the designated quantitation laboratory. (Questions regarding packaging may be resolved with the designated quantitation laboratory).
   3. If quantitation is requested after analysis has been completed and the evidence has been returned, the laboratory will advise the requestor to resubmit the evidence directly to the designated quantitation laboratory.

D. For exhibits greater than or equal to 1 kilogram:
   1. The receiving laboratory will perform qualitative analysis, including an attempt to determine the salt form, and collect representative samples using the following guidelines:
      a) For a one item exhibit:
         i. Take approximately 1 gram samples from 15 representative locations from the exhibit and combine into one sample.
         ii. The location of sampling on evidence in solid form, such as compressed bricks, shall be documented in the case record.
            • For example, for a one brick exhibit a description may include – samples taken from each of the 8 corners, from the center core, and ~¼” deep in 3 areas on the top and bottom surfaces. A sketch or digital image with notes would also meet the requirement.
            • For example, for a bag of powder – samples are taken after shaking. Location of sampling on the evidence is understood and not required to be documented.
      b) For multi-item exhibit:
         i. Take samples of at least 1 gram from each item until a total minimum of 15 grams is obtained. Combine the individual samples into one composite sample.
            Note: This option must be chosen if less than ten items are present. If greater than ten items are present, either this method or ii. below may be applied.
            OR
ii. Follow the statistical sampling plan at 90% in SD-03-01 to identify the number of samples required for quantitation. Use the Random Number Generator (RNG) (see section 7) to select which items to sample.

- The samples selected may include samples not analyzed during qualitative analysis.
- Retain a copy of the RNG results in the case record.
- Take approximately 1 gram samples from each item selected by RNG. A total minimum of 15 grams must be obtained. Combine the individual samples into one composite sample.
- The items from which the samples were collected and the location of sampling on evidence in solid form, such as compressed bricks, shall be documented in the case record.

2. In LIMS, sub-itemize the composite sample into a separate container (LIMS-11-05).

3. Forward the composite sample to the designated quantitation laboratory.

   **Note:** The composite sample for quantitation is considered evidence and will be tracked in LIMS.

4. The receiving laboratory should retain the bulk evidence until the quantitation sample has been received back from the quantitation laboratory.

### 6.2 Instructions for Quantitation Laboratories

#### A. Any cases submitted to a laboratory for quantitative analysis must have all DPS seals intact.

#### B. Exhibits greater than or equal to 5 grams but less than 1 kilogram:

1. For a one item exhibit:
   a) **For exhibits with a net weight less than 30 grams, homogenize the entire exhibit.**
   b) **For exhibits with a net weight greater than or equal to 30 grams but less than 1 kilogram:**
      i. Take approximately 1-gram samples from 15 representative locations from the exhibit. Combine into one sample and homogenize for analysis.
      ii. The location of sampling on evidence in solid form, such as compressed bricks, shall be documented in the case record.

   - **For example,** for a one brick exhibit a description may include – samples taken from each of the 8 corners, from the center core, and approximately 1⁄4" deep in 3 areas on the top and bottom surfaces. A sketch or digital image with notes would also meet the requirement.
   - **For example,** for a bag of powder – samples are taken after shaking. Location of sampling on the evidence is understood and not required to be documented.

2. For multi-item exhibit:
   a) **For exhibits with a net weight less than 30 grams, homogenize the entire exhibit.**
   b) **For exhibits with a net weight greater than or equal to 30 grams but less than 1 kilogram:**
      i. Take samples of at least 1 gram from each item until a total minimum of 15 grams is obtained. If the exhibits are less than 1 gram each, take approximately 0.2 grams each until a minimum total of 15 grams. Combine into one sample and homogenize for analysis.

   **Note:** This option must be chosen if less than ten items are present. If greater than ten items are present, either this method or ii. below may be applied.

   **OR**
ii. Follow the statistical sampling plan at 90% in SD-03-01 to identify the number of samples required for quantitation. Use the Random Number Generator (RNG) (see section 7) to select which items to sample.

- The samples selected may include samples not analyzed during qualitative analysis.
- Retain a copy of the RNG results in the case record.
- Take samples of approximately 1 gram from each item selected by RNG. A minimum total of 15 grams must be obtained. If the exhibits are less than 1 gram each take approximately 0.2 grams each until a minimum total of 15 grams. Combine into one sample and homogenize for analysis.
- The items from which the samples were collected and the location of sampling on evidence in solid form, such as compressed bricks, shall be documented in the case record.

C. Samples Collected from Exhibits Greater than or Equal to 1 Kilogram shall be processed as follows:

D. Verification of Equipment and Homogenization

1. One method blank will be prepared for the mortar and pestle.
   a) The blank will be run using the same method and instrument as the quantitation sample(s).
   b) Evaluation and documentation of the blank will be done in accordance with the relevant instrument policy

2. Homogenize the quantitation samples separately using the mortar and pestle.

3. Proceed to a quantitative analysis method.

E. Evidence Return

1. Return quantitation evidence to the laboratory that originally performed the qualitative analysis.

2. Return quantitation evidence to the submitting agency if received directly from that agency.

7 Random Number Generator (RNG) Instructions

All containers / items in an exhibit must be numbered prior to applying random number generator.

1. Open the Random Number Generator Excel file (LAB-SD-30) and enter the case number and analyst information.

2. Enter the total number of units in the exhibit and the total number of units to be selected (from the SSP at 90%).

3. Any time the RNG is used in casework, the results shall be retained in the case record. The results of the RNG should be immediately preserved by printing on paper or saving in electronic format.

4. After obtaining the RNG values, pressing enter or reopening the file will generate a new random number.

5. Identify the units to be sampled by following a row across on the printout starting at the upper left corner.

6. If a number is repeated (i.e., unit is already selected) skip and continue to the next number.
8 Advantages
   A. Random selection process is used to ensure all units in a population have an equal chance of being selected and avoid selection bias.
   B. Random selection allows the use of statistical methods to analyze sample results and allows inferences to be made on the whole population.

9 Records
Seized Drugs Worksheet (in LIMS or LAB-SD-01)
Random Number Generator (LAB-SD-30)

10 Literature References and Supporting Documentation
08 IDENTIFICATION OF SUBSTANCES OTHER THAN DRUGS

SD-08-01 RESORCINOL TEST

1 Scope
Resorcinol test is a chemical reactivity test that may be performed to determine the possible presence of sugar. Infrared spectroscopy or X-ray diffraction may be used to provide positive identification if the evidence permits.

2 Related Chapters
Fourier Transform Infrared Spectrophotometry (SD-06-02)
Modified Molisch Test (SD-08-02)

3 Safety
A. Chemical spot tests may use a variety of corrosive, caustic, or other dangerous chemicals. Caution should always be practiced and appropriate personal protective equipment used.
B. Refer to SDS for additional safety information for specific chemicals.

4 Equipment and Materials
- Test tubes, pipettes, or other appropriate containers/items
- Resorcinol
- Concentrated Sulfuric Acid

5 Standards, Controls, and Calibration
A. Freshly prepared reagent will be performance checked with sucrose and the results recorded in a retrievable logbook.
B. Performance of reagents will be checked monthly and the results of the checks placed in a logbook.
C. If the reagent has not been used for a month or more, it must be checked using a standard (and the results of the check logged) before its use with case samples.
D. The expected result of the performance check is an orange to red color at the interface.

6 Procedure
6.1 Preparation Resorcinol Reagent, 0.1% (w/v)
   1. Dissolve 0.1 g Resorcinol in 100 mL water.
   2. Performance check reagent.

6.2 Test Procedure
   1. Dissolve approximately 20 mg sample in 1 mL Resorcinol reagent.
   2. Stratify the sample mixture on top of approximately 2 mL Sulfuric Acid. Do not mix.
   3. Record any resulting color reaction(s).
7 Interpretation

A. A reaction which forms an orange to red color at the interface indicates the possible presence of a carbohydrate (sugar).

B. Sucrose, Lactose, d-Fructose, Maltose, and Dextrose (d-Glucose) will produce a positive result.

8 Literature References and Supporting Documentation

SD-08-02 MODIFIED MOLISCH TEST

1 Scope
This test is a chemical reactivity test that may be performed to determine the possible presence of carbohydrates, which is similar in application to the Molisch test. Infrared spectroscopy or X-ray diffraction may be used to provide positive identification if the evidence permits.

2 Related Chapters
Fourier Transform Infrared Spectrophotometry (SD-06-02)
Resorcinol Test (SD-08-01)

3 Safety
A. Chemical spot tests may use a variety of corrosive, caustic, or other dangerous chemicals. Caution should always be practiced and appropriate personal protective equipment used.
B. Use a glass Pasteur pipette to add the H$_2$SO$_4$; do not use a mechanical pipettor with concentrated acids.
C. Refer to SDS for additional safety information for specific chemicals.

4 Equipment and Materials
- Test tubes, pipettes, or other appropriate containers/items
- 1-naphthol
- Ethanol
- Concentrated Sulfuric Acid

5 Standards, Controls, and Calibration
A. Freshly prepared reagent will be performance checked with sucrose and the results recorded in a logbook.
B. Performance of reagents will be checked monthly and the results of the checks placed in a logbook.
C. If the reagent has not been used for a month or more, it must be undergo a performance check using a standard (and the results of the check logged) before it is used with case samples.
D. The expected result for a performance check is an orange to red color at the interface.

6 Procedure
6.1 Preparation of Modified Molisch Reagent 15% (w/v)
1. Dissolve 1.5 g 1-naphthol in 10 mL ethanol.
2. Store the reagent, protected from light, at room temperature.

6.2 Test Procedure
1. Dissolve a portion of the sample in a minimum of deionized water in a test tube.
2. Place two drops of the test solution in a spot plate, 1 drop 15% 1-Naphthol reagent and 2 drops Sulfuric Acid. (Reminder: Always add acid to water.)
3. Record any resulting color reaction(s).
7 Interpretation

A. A reaction which forms a blue to red-violet color at the interface indicates the possible presence of carbohydrates (sugar).

B. This test may detect compounds other than carbohydrates; however a negative result indicates the absence of carbohydrates.

C. Different sugars may give slightly different colors. Starch may also give a positive reaction.

8 Literature References and Supporting Documentation


SD-08-03  ACIDIC SOLUTIONS

1  Scope
Analysis of acidic solutions in order to presumptively determine the presence of chloride indicative of hydrochloric acid and/or sulfate indicative of sulfuric acid.

2  Related Chapters
Iodine and Hydriodic Solutions (SD-08-04)

3  Safety
A. Chemical spot tests may use a variety of corrosive, caustic, or other dangerous chemicals. Caution should always be practiced, and appropriate personal protective equipment used.
B. Refer to SDS for additional safety information for specific chemicals.
C. This procedure should be performed in a fume hood.

4  Equipment and Materials
- Spot plates, pipettes, or other appropriate containers/items
- pH indicating strips (range of 0-14)
- ~1% Barium chloride (BaCl₂) Reagent
- ~5% Silver nitrate (AgNO₃) Reagent
- Concentrated Nitric acid
- Concentrated Ammonium hydroxide

5  Procedures
5.1  pH determination
1. Remove an aliquot of the liquid and transfer to a culture tube.
2. Test the liquid with pH paper.
3. Record pH if any indicated.
4. Test the vapor above the liquid with pH paper.
5. Record the pH of vapor (fuming acids) above liquid if any indicated.

5.2  Miscibility determination
1. Mix the sample with an approximately equal volume of water and agitate.
2. Record any observations.
3. If the liquid forms two layers, it indicates that an organic solution may be present and other tests may be performed.
4. If the liquid forms one layer, it may indicate that an aqueous solution is present; proceed with anion identification.
5.3 Anion identification

The following procedures are used to identify anionic components of an acidic unknown. See also the Acid Analysis Flow Chart (LAB-SD-03).

A. Silver Nitrate (AgNO₃) Test
   1. Prepare reagent
      a) Dissolve 0.5 g silver nitrate in 10 mL H₂O.
      b) Keep tightly capped and store at room temperature.
      c) Quality test reagent with aqueous NaCl; a white precipitate should form.
   2. Add a few drops of Silver Nitrate reagent to a dilute solution of the liquid unknown.
   3. Record any observations.
   4. If one of the following conditions exists, proceed to the appropriate test:
      a) If a white precipitate forms, proceed to HNO₃ Solubility Test.
      b) If a yellow precipitate forms, proceed to NH₄OH Solubility Test.
      c) If no precipitate forms, proceed to Barium Chloride Test.
      d) If a black or other precipitate forms, testing may be stopped.

B. Nitric Acid (HNO₃) Solubility Test
   For sample which formed a white precipitate in the Silver Nitrate Test
   1. Decant the liquid and retain the white precipitate from the Silver Nitrate Test.
   2. Add several drops of concentrated nitric acid to the white precipitate.
   3. Record any observations.
   4. If the white precipitate does not dissolve, proceed to NH₄OH Solubility Test.

C. Ammonium Hydroxide (NH₄OH) Solubility Test
   For sample which formed a yellow precipitate in the Silver Nitrate Test and did not dissolve in Nitric Acid Solubility Test
   1. Add a few drops of Silver nitrate reagent to a fresh dilute aliquot of the liquid unknown.
   2. Decant the liquid and retain the yellow precipitate.
   3. Add several drops of concentrated NH₄OH to the yellow precipitate.
   4. Record any observations.

D. Ammonium Hydroxide (NH₄OH) Solubility Test
   For sample which formed a yellow precipitate in the Silver Nitrate Test
   1. Decant the liquid and retain the yellow precipitate.
   2. Add several drops of concentrated NH₄OH to the yellow precipitate.
   3. Record any observations.
E. Barium Chloride (BaCl₂) Test

1. Prepare reagent
   a) Dissolve 0.1 g barium chloride in 10 mL H₂O.
   b) Keep tightly capped and store at room temperature.
   c) Quality test reagent with aqueous Na₂SO₄; a white precipitate should form.

2. Add a few drops of Barium Chloride reagent to a fresh dilute aliquot of the liquid unknown.

3. Record any observations.

6 Interpretation

6.1 Anion Interpretation

A. The following information must be considered when interpreting results of the anion identification procedure.

1. Silver Nitrate Test
   a) White precipitate - indicates chloride or carbonate ion
   b) Yellow precipitate - indicates chloride, iodide, or bromide ion
   c) No precipitate - indicates possible sulfate, nitrate, acetate, or phosphate ion
   d) Other precipitates that are formed are considered to be negative

2. Solubility in concentrated HNO₃
   a) White precipitate dissolves - indicates carbonate ion
   b) White precipitate does not dissolve - indicates possible chloride ion

3. Solubility in concentrated NH₄OH
   a) White precipitate dissolves - indicates chloride ion
   b) White precipitate does not dissolve - indicates another ion may be present
   c) Yellow precipitate dissolves - indicates chloride ion
   d) Yellow precipitate does not dissolve - indicates possible iodide ion may be present, proceed to Iodine and Hydriodic Solutions (SD-08-04)

4. Barium Chloride Test
   a) Precipitate - indicates sulfate ion
   b) No precipitate - indicates other ions may be present

6.2 Acid Interpretations

A. An acidic pH in conjunction with presumptive anion identification gives presumptive identification of various acids.

1. Indications for the presence of Hydrochloric Acid
   a) Silver Nitrate Test forms white precipitate
   b) White precipitate does not dissolve in concentrated HNO₃
   c) White precipitate dissolves in concentrated NH₄OH
d) May be confirmed by vapor phase FTIR analysis or by FTIR analysis of a KBr pellet of the white precipitate from the Silver Nitrate Test to confirm AgCl.

2. Indications for the presence of Sulfuric Acid
   a) No precipitate forms with the Silver Nitrate Test
   b) Barium Chloride Test forms precipitate

3. If neither Hydrochloric Acid nor Sulfuric Acid is indicated, then the conclusion is that an unknown acid is present. It may be necessary to proceed to determination of Iodine or Hydriodic Acid solutions.

B. Other Interpretations

The measurement of acidic vapors above a sample of unknown liquid may indicate a concentrated acid solution where the pure compound is a vapor at room temperature and pressure.

7 Limitations

A. A pH >5 in conjunction with presumptive anion identification may be used to presumptively identify neutralized solutions or neutral salts in solution.

B. The procedures may give incorrect or misleading results if more than one anion is present in solution.

8 Records

Seized Drugs Worksheet (LAB-SD-01)

Acid Analysis Flow Chart (LAB-SD-03)

9 Literature References and Supporting Documentation

Whipple, O.K. Chemical properties and identification of ions.

CRC Handbook of Chemistry and Physics

DPS Internal Validation
SD-08-04  IODINE AND HYDRIODIC SOLUTIONS

1  Scope
This document addresses issues related to iodine solid and/or liquids containing iodine. Iodine is often converted to hydriodic acid (iodide in acidic solution) during the manufacture of methamphetamine. Iodine may be found in liquid samples with or without hydriodic acid such as tincture of iodine.

2  Related Chapters
Acidic Solutions (SD-08-03)

3  Safety
A. Iodine is a toxic substance that sublimes at room temperature.
B. Use caution when handling any unknown substance or chemical.
C. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
D. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

4  Equipment and Materials
• Culture tubes
• Disposable pipettes
• pH indicating strips (range of 0-14).

5  Procedures
5.1  Solid Sample
A. Document the physical appearance of the sample.
B. Volatility test
   1. Place a small amount of the sample into a culture tube.
   2. Add small piece of white paper and cap tube.
   3. Record any observations.
C. Solubility test
   1. Place a small amount of the sample into a culture tube.
   2. Add deionized water to the tube.
   3. Add chloroform to the tube.
   4. Record any observations.
   5. Proceed to Acidic Solutions (SD-08-03) with the solution.
5.2 Liquid Sample

A. Determine the pH, miscibility, and anion identification from *Acidic Solutions (SD-08-03)*.

B. Chloroform Test
   1. Add an aliquot of the sample to a culture tube.
   2. Dilute with deionized water, if necessary.
   3. Add chloroform.
   4. Record any observations.

6 Interpretation

6.1 Solid Sample

Consider the following information when interpreting results of the presumptive identification of the presence of solid iodine:

A. Iodine appears as a silver/grey metallic flake or pellet.
B. Solid iodine sublimes to a vapor and discolors paper.
C. Iodine is only slightly soluble in water.
D. A purple color in the chloroform layer indicates the presence of iodine.

6.2 Liquid Sample

Consider the following information when interpreting results of the presumptive identification of the presence of iodine/iodide:

A. A purple color in the chloroform layer indicates the presence of iodine.
B. A low pH (0-4) indicates acidic solution.
C. Anion Interpretation based on results from *Acidic Solutions (SD-08-03)* where the formation of a yellow precipitate in the Silver Nitrate Test which is not soluble in NH₄OH indicates presence of iodide ion

7 Records

Seized Drugs Worksheet (LAB-SD-01)
Acid Analysis Flow Chart (LAB-SD-03)

8 Literature References and Supporting Documentation

Whipple, O.K. Chemical properties and identification of ions.
CRC Handbook of Chemistry and Physics
DPS Internal Validation
SD-08-05 RED PHOSPHORUS

1 Scope
Red phosphorus is listed in the HSC 481.124. It may be used in clandestine manufacturing of methamphetamine via reduction of ephedrine or pseudoephedrine utilizing hydriodic acid or iodine.

2 Related Chapters
Acidic Solutions (SD-08-03)
Iodine and Hydriodic Solutions (SD-08-04)

3 Safety
A. Red phosphorus is a slightly toxic, flammable powder that may cause burns upon skin contact.
B. Use caution when handling any unknown substance or chemical.
C. For hazardous materials or possible hazardous materials, use appropriate personal protective equipment.
D. Safety Data Sheets are available in the laboratory if additional information is needed about a chemical.

4 Equipment and Materials
- Porcelain plate
- Disposable glass pipettes
- pH indicating strips (range of 0-14).
- Safety match

5 Procedures
5.1 Friction Test
*Note:* Perform this test in a fume hood.
1. Place a small amount of the sample onto a porcelain plate.
2. Strike a “Safety” match through the powder.
3. Record any observations.

5.2 Ignition Test
*Note:* Perform this test in a fume hood.
1. Place a small amount of the sample on the tip of a metal spatula.
2. Expose to a flame.
3. Record any observations.
5.3 Flame Test

Note: Perform this test in a fume hood.

1. Place a small plug of glass wool into the barrel of a glass pipette and tamp down.
2. Place a small amount of the sample into the barrel of the pipette followed with a small plug of glass wool.
3. Place a rubber bulb on end of the pipette.
4. Apply a flame to the area of the pipette containing the sample.
5. (optional) With a pH strip moistened with water, check the pH of the fumes that evolve from the end of pipette.
6. Use the rubber bulb to force air through the pipette.
7. Record any observations.

6 Interpretation

Consider the following information when interpreting results for the presumptive presence of red phosphorus:

A. Red phosphorus will ignite with the friction of a “Safety” match.
B. On ignition, phosphorus produces a yellow-orange flame and white smoke.
C. The flame may be accompanied by yellow acidic fumes (pH < 7).

7 Records

Seized Drugs Worksheet (in LIMS or LAB-SD-01)

8 Literature References and Supporting Documentation

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