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Preparer

For Ruben A. Rendon, Jr. by Forrest W. Davis Date: 03/03/2009
Controlled Substance Advisory Board Chair

Concurrence

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Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
01	12/01/2002	Minor Revision Deletion Evidence Handling Reverse-Role Moved to Section 3 Marihuana, Peyote, and Mushroom exam/preparation Addition Section 3Derivatization LSD Moved to Section 4 Chemical screening spot tests Rename Scott to Cocaine Test Addition Section 4 sulfuric acid, formaldehyde-sulfuric, Liebermann, and phosphorus tests Addition Section 5 silver-copper nitrate test
02	07/01/2003	Minor Revision Addition Section 2 LAB-CS-01 and LAB-CS-02 Addition Section 3 Derivatization GHB
03	12/01/2003	Modification to title CS-02-01A “Approved List of Reference Abbreviations” Deletion Section 2 LAB-CS-01 and LAB-CS-02 Addition Section 4 CS-04-16, CS-04-17
04	01/01/2005	Addition of CS-04-18 Janovsky Test Move CS-09-01 to CSR major revision Rename CS-09 group to “Identification of Substances Other than Drugs” Addition CS-09-01 Resorcinol Test Addition CS-09-02 Modified Molisch Test

Version #	Effective Date	Brief Description of Change(s)
05	05/01/2005	Addition CS-02-01A Standard Abbreviations List Addition CS-03-01A Controlled Substances Worksheet Instructions Addition CS-07-06 HPLC Addition CS-08-03 HPLC Quantitation Rename CS-04-03 Cobalt Thiocyanate (Cocaine) Tests
06	10/10/2006	Addition CS-01-03 Controlled Substance Overview Addition CS-03-08 Germination of Marihuana Addition CS-09-03 Acidic Solutions Addition LAB-CS-03 Acid Analysis Flowchart Addition CS-09-04 Iodine and Hydriodic Solutions Addition CS-09-04 Red Phosphorus Addition Forms
07	07/20/2007	Revised: CS-01-01, CS-02-01, CS-02-02, CS-03-01, CS-07-01, CS-07-03
08	08/21/2007	Revised: CS-01-01
09	05/05/2008	Revised: CS-01-01, CS-02-02, CS-03-01, CS-04-18, CS-07-03, CS-07-04
10	02/09/2009	Revised: CS-01-01, CS-02-01, CS-02-01A, CS-02-02, CS-03-02, CS-04-03, CS-04-12, CS-06-01
11	03/03/2009	Revised: CS-03-02A

REPORTING GUIDELINES

1 Scope

To establish standards for reporting the results from the analysis of controlled substances, dangerous drugs, clandestine laboratory chemicals and other substances 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* which contains the *Texas Controlled Substances Act*. The law determines the terminology used in reporting the identification of most controlled substances and requires the net 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"***, unless the substance has been quantitated. Then, report the quantitation results as percent by weight for solid samples or as weight per volume or percent weight per weight for liquid samples. Marihuana and Peyote will not be preceded with "contains" unless they contain other materials.

3. If more than one controlled substance is identified in a sample, report the names in alphabetical order after ***"Contains"***, unless the controlled substances are quantitated. Report all of the controlled substances identified.

   Examples: *Contains Amphetamine and Methamphetamine*

   Contains Cocaine and Phencyclidine

4. If the sample is quantitated, report the results in parenthesis after the name of the substance.

   Examples: *Cocaine (10%) and Methamphetamine (10%)*

   Methamphetamine (1.6 mg/mL)

5. 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) and note the presence of the dangerous drug(s) on the worksheet. At the discretion of the analyst, it may be necessary to report other substances identified for certain cases.

6. If a sample contains only dangerous drugs, report all dangerous drugs identified. Report them using their common generic drug name, not their pharmaceutical trade name.

   Example: Contains _[generic drug name]_

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

1. Quantities of substance(s) reported based on pharmaceutical information must include a footnote, such as:

   "Information from the pharmaceutical company indicates that this preparation contains…"

   "Pharmaceutical identification indicates…"

2. Quantities of controlled substance(s) reported with other relevant substances based on analytical determination must include an appropriate footnote used, such as:

   "The quantity of controlled substance, or any of its salts, is not more than the amount categorized for this substance in penalty group 3 [or 4], and contains other associated ingredients commonly found with a pharmaceutical preparation."

C. Reporting Marihuana, Marihuana Seeds and Hashish

1. Report plant substance identified as marihuana as ***"Marihuana"*** and report the weight.

2. If a significant amount of an impurity, such as tobacco, is present in the marihuana sample, make a conservative visual or microscopic estimate of the percent of marihuana present and report the net weight. Report substance beginning with **"*Contains"*** if the sample does not consist entirely of marihuana.

   Example: *Contains Marihuana*

   or, if the percentage of marihuana is estimated;

   Marihuana (33%)

3. Report the results of marihuana pipes and the charred remains of marihuana as **"*Marihuana"*** and the weight as **"*trace" only if*** microscopically identifiable marihuana is present. If insufficient physical characteristics are present to identify marihuana, then the results may be reported as inconclusive.

4. For cases that consist of marihuana seeds only, they may be reported as **"*Marihuana seeds"*** and the weight in grams and/or ounces. If germination is attempted and no seeds germinate, report as negative with a footnote: "Marihuana seeds were identified and determined to be incapable of beginning germination".

5. Report hashish and liquid extracts as **"_Contains Tetrahydrocannabinol_"** and the weight in grams.

D. Reporting Peyote Samples

For plants visually identified as *peyote* and analyzed to confirm the presence of mescaline, report as **"_Peyote_"** with the weight in grams. If the plant material cannot be visually identified as *peyote* or it is a powdered sample, report as **"_Contains Mescaline_"**, unless quantitated, along with the weight in grams.

E. Reporting Mushroom Samples

Report psilocybin mushrooms as **"_Contains Psilocin_"**. Psilocybin may be reported if it has been identified using TLC and FTIR or TLC and a derivative procedure on GC/MS.

F. 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 Weights and Volumes

1. If a substance is identified in a powdered sample and the results are to be reported, report the weight of powdered sample.

2. Report the weight of liquid samples, if a controlled substance or dangerous drug is identified. The volume may also be reported.

3. If the contents are identified and reported, include the weight of tablets and capsules on the report. If desired, the number of tablets and capsules also may be reported.

4. Except for marihuana, report the net weight in grams, if the sample ranges from 0.01 gram to 1,000 grams. Weights greater than or equal to 1,000 grams should be reported as ***kilograms***. Weights less than 0.01 gram may be reported as ***trace***.

5. For marihuana samples weighing less than one pound, report the weight of marihuana in grams and/or ounces. Report marihuana samples weighing more than one pound in pounds to at least one decimal place. 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.

[To convert grams to ounces, use 28.4 grams per one ounce.]

2.3 Reporting Abuse Units

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

2.4 Miscellaneous

1. Dilutants (diluents) and adulterants should not be reported on a routine basis. However, they may be reported at the discretion of the analyst, if requested by the submitting official or prosecutor's office or if it is deemed necessary due to case circumstances.

2. The salt form of the drug will not be reported unless that salt form has been properly identified using FTIR or other scientifically accepted procedures. Likewise, the base form will not be reported unless the base form has been verified using FTIR or other scientifically accepted procedures.

3. For pharmaceutical preparations containing dextropropoxyphene, if the stereoisomer is determined by pharmaceutical information, then a footnote must be used, such as:

   "Information from the pharmaceutical company indicates the dextrorotatory isomer."

4. 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*

5. 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 and weight.

6. If an insufficient sample is present for a positive identification of a controlled substance or dangerous drug but one is suspected by a positive preliminary test or instrumental analysis, report as **"*Inconclusive**"** with a footnote stating: **"*Insufficient sample for positive identification.*"**

7. Exhibits that are not analyzed are reported as **"*No Analysis*"** or ***"No Analysis Requested"*** and no weight is reported. Appropriate footnotes may be added at the analyst's discretion, such as:

   "In view of the other exhibits, no examination was performed on this exhibit. If analysis is required for prosecution or other purposes, please notify this laboratory."

   "Pharmaceutical information indicates that no controlled substance is present."

8. Samples may be reported as **"*Negative*"** only after the sample has been subjected to sufficient analytical examinations. No weight will be reported and an appropriate footnote may be chosen at the discretion of the analyst.

9. Occasionally substances that are not controlled substances or dangerous drugs may be analyzed and reported. If the substance has not been confirmed, the substance will be reported as either **"*", "Negative", or "No Analysis"** followed by a footnote.

    a) *Exceptions to reporting weight or volume include anhydrous ammonia or water samples containing ammonia, red phosphorus, lithium, and iodine samples. No weight is reported, although it can be added to the footnote, if desired.*

    b) *Appropriate footnotes may include:* ***"No controlled substance was detected. The appearance of the sample and the results of presumptive test(s) indicate that this exhibit contains [substance]"****.*

10. Other footnotes may be added to the report at the discretion of the analyst when circumstances mandate it. Additional customized footnotes may be added to the standardized footnote list at the discretion of the laboratory supervisor or manager.

Preparer

Ruben A. Rendon, Jr. Date: 11/21/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 01/22/2009
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
01	12/01/2002	Minor Revision, Documentation and record keeping text moved to separate document Modification Section 2.1 B 5 footnote for when none of the seeds germinate "Marihuana seeds were identified and determined to be incapable of beginning germination". Addition Section 2.3, 5 requiring a positive preliminary test or instrumental analysis
02	07/01/2003	Minor Revision with respect to reference to IR changed to FTIR Modification Section 2.1 A. 5. changed "chemical" to "generic drug" Deletion Section 2.1 B. 3. Moved to CS-03-01 Modification Section 2.1 B. 3. From "Contains marihuana residue" to "marihuana"… "only if,…" "If insufficient physical characteristics are present to identify marihuana, then the results may be reported as inconclusive." Deletion Section B. 6. Moved to CS-03-01 Deletion Section B. 7. Moved to CS-03-01 Deletion Section 2.2 1. Addition Section 2.3 "Reporting Abuse Units" from Section 2.2 7. Deletion Section 2.2 8. Moved to Section 2.4 miscellaneous section Modification Section 2.4 9. "…that are not controlled substances or dangerous drugs may be analyzed and reported. If the substance has not been confirmed, the substance will be reported as either **"*", "Negative", or "No Analysis"** followed by a footnote. Modification Section 2.4 9a. "Exceptions to reporting weight or volume include anhydrous ammonia or water samples containing ammonia, red phosphorus, lithium, and iodine samples…

Version #	Effective Date	Brief Description of Change(s)
02	07/01/2003	*Addition Section 2.4* 9b. "Appropriate footnotes may include: **"No controlled substance was detected. The appearance of the sample and the results of presumptive test(s) indicate that this exhibit contains [substance]"**."
03	12/01/2003	Modification to Section 2: "DPS reporting guidelines are based on the laws and definitions provided in Chapters 481-485 of the *Texas Heath and Safety Code…"* Addition Section 2.1 A #6 When the following conditions have been met, dangerous drugs may be reported as "*No Analysis (generic drug name)**" with the footnote "*Pharmaceutical identification only. No chemical analysis performed.*" *a) Offense is listed as "Possession"* *b) Identified by Pharmaceutical ID and no analytical data is available* Addition to Section 2.1 B #4 "If germination is attempted and no seeds germinate…." Modification to Section 2.4 #5 "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 and weight."
04	10/10/2006	Deletion Section 2.1 A #6 regarding reporting dangerous drugs by pharmaceutical identification Addition Section 2.4 #4 "For pharmaceutical preparations containing dextropropoxyphene, if the stereoisomer is determined by pharmaceutical information, then a footnote must be used, such as: **"Information from the pharmaceutical company indicates the dextrorotatory isomer."**"
05	07/20/2007	Addition Section 2.1, A: The reports shall indicate that a portion of the evidence was analyzed, such as "A sampling was utilized in the analysis to represent the entire amount."
06	08/21/2007	Deletion Section 2.1, A: ~~The reports shall indicate that a portion of the evidence was analyzed, such as "A sampling was utilized in the analysis to represent the entire amount."~~
07	05/05/2008	Major revision – Sections 2.1 and 2.4 Advisory Board 02/27/2008
08	02/09/2009	Major revision – Sections 2.1 and 2.4 Advisory Board 11/20/2008

CASE DOCUMENTATION

1 Scope

These policies are established as minimum requirements for additional case documentation and record-keeping required for controlled substance cases.

2 Contents of Case Folder

A. Evidence Record Sheet

B. Laboratory Submission Form

C. Notes containing additional information about the evidence, analysis procedures or any other explanatory notes may be recorded on separate sheets. On each page include the case number, analyst's handwritten initials and the date recorded. Page numbers may be added.

D. Analytical data

1. All charts, spectra, notes and photographs will be maintained and archived in accordance with the Case documentation section of the Laboratory Operations Guide.

2. If solvent blanks were run on GC/MS prior to a trace sample, then the charts will be maintained in the case folder.

3 Exam Counting Guidelines

A. Examinations for drug analysis casework must be counted and recorded on the worksheet.

1. Number Received 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 = 25).

2. Number Analyzed equals the actual number of individual items that were analyzed. However, pharmaceutical identification of 500 tablets is one (1) for Analysis and one (1) for Number Analyzed, not 500. Also, the examination of a sheet of 250 LSD squares under UV light is equal to one (1) for Number Analyzed, not 250, and is equivalent to one (1) for Analysis.

3. Each spot test, instrumental examination, determination of net weight, and microscopic or visual examination shall be counted as one (1) examination each. Per exhibit, preliminary pharmaceutical examinations will be counted as one (1) examination only, no matter how many dosages are received or how many pharmaceutical references are searched. For example, if 500 tablets are received in a particular exhibit, only one (1) pharmaceutical examination can be counted and if several references are used, only one (1) pharmaceutical examination can be counted.

4. Number of Examinations refers to the total number of examinations performed. Add the number of spot tests run to the number of microscopic exams to the number of instrumental analyses, etc.

B. When evidence is re-examined by another analyst and that analyst prepares a separate work sheet, the examinations performed on the evidence are counted in the monthly statistics as for other cases.

Preparer

Larry Todsen Date: 04/24/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest Davis Date: 04/24/2006
Quality Assurance

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00	12/01/2002	Original Issue, Minor Revision, Text from Chapter 1 Deletion from Section 2 D 2 "…or in a retrievable form such as a quality control folder or electronic format." Addition to Section 3 B "…determination of net weight…." counted as an examination. Deletion Literature and Supporting Documentation
01	07/01/2003	Deletion Section 3 C Monthly Controlled Substance Report
02	12/01/2003	Modification to Section 3 "Statistics for drug analysis casework must be counted…." Addition Section 3 A #1 Number Received Addition Section 3 A #2 Number Analyzed Deletion to Section to 3 A #4 counting controls Deletion Section 3 B #2 quantitation counting as one exhibit
03	01/01/2005	Modification to Section 3 A "Examinations ~~Statistics~~ for drug analysis…."
04	10/10/2006	Modification to Section 3 A #3 "…Per exhibit, preliminary pharmaceutical ~~identification~~ examinations will be counted as one (1) examination only, no matter how many dosages are received or how many pharmaceutical references are searched. For example, if 500 tablets are received in a particular exhibit, only one (1) pharmaceutical ~~test~~ examination can be counted and if several references are used, only one (1) pharmaceutical examination can be counted."

Appendix A—Approved Standard Abbreviations List

1.1 Color

B, b	blue
BLK, blk	black
BRN, brn	brown
GRN, grn	green
O, o	orange
P, p	purple
PK, pk	pink
R, r	red
V, v	violet
WHT, wht	white
Y, y	yellow

1.2 Other

comp	composite, composition, or combination
→	contains, containing, placed inside, into
cryst	crystal(s); crystalline
dk	dark
env	envelope
evid	evidence
ex	exhibit
insuf	insufficient
lt	light
NA	not analyzed, no analysis
PDMABA	para-dimethylaminobenzaldehyde
pharm	pharmaceutical identification
PS	plant substance
pl	plastic
Rx	prescription
subst	substance
wk	weak

Preparer

Andrew Macey Date: 04/04/2005
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 04/04/2005
Quality Assurance

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00	05/01/2005	Original Issue

CONTROLLED SUBSTANCE OVERVIEW

1 Routine Examination

A. Routine drug identification may include any or all of the following at the discretion of the laboratory:

1. Determine net weight, and perform chemical screening examinations, instrumental confirmation tests, pharmaceutical identification of tablets/capsules, and analysis of materials related to lab seizures.
2. Some exhibits may not be analyzed depending on circumstances of the case
3. Possibly photograph and/or repackage evidence
4. DPS evidence submissions of excess quantity cases will follow laboratory policies for destruction
5. Quantitation for cases containing > 2 kg powder or > 4 L liquid
6. Reexamination for QA purposes or court purposes. However, evidence previously examined by another laboratory will not be reexamined without authorization by the Director of the Crime Laboratory Service.
7. Syringes typically are not examined without a request from the prosecutor's office.
8. Laboratories do not provide services for destruction of hazardous materials, clandestine laboratory clean-up, etc.

2 Examiner Approval

Demonstration of competency in the use of controlled substances standard operating procedures is required prior to independent casework. The following areas require director approval to allow independent casework by an analyst:

1. Marihuana
2. Controlled Substances

3 Proficiency

A. The areas in the controlled substances discipline for which individual proficiency testing is currently available include:

1. Controlled Substances (External Test, Internal Test, and/or Case Reexamination)
2. Marihuana (Internal Test, and/or Case Reexamination)

Preparer

Larry Todsen Date: 06/12/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 06/12/2006
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00	10/10/2006	Original Issue

STANDARDS AND REFERENCES

1 Scope

These policies serve to establish guidelines for the use of drug reference samples and libraries.

2 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 the name of the drug, the date received or used, the initials or name of the person making the entry in the log, the source from which the drug was obtained, the purpose for which it was used, the lot or identification number if available, the form or concentration, the amount, the balance remaining, and the disposal date.

B. The quantity of reference drugs to be kept 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 to them.

D. 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.

E. Internal reference standards should be thoroughly analyzed and characterized before use as standards or controls.

3 Quality Control Procedures for Drug Standards

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

B. Some commercially prepared drug standards are mailed with GC/MS and other quality control data. These data sheets will be retained.

4 Verification of Standards That Cannot Be Purchased Commercially

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 (i.e. case sample) or obtained from another laboratory.

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

5 Source References

A. When analyzing compounds, particularly drugs, 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 file.

B. References used for Pharmaceutical ID will be documented in case file.

C. The Approved List of Reference Abbreviations (Appendix A) will be used to denote the common references for standard spectra. Addition of other routine references may be added with the approval of the Controlled Substances Advisory Board.

6 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 evidentiary substances:

1. The compound used to generate the spectrum must be traceable. The person that generates the spectrum must note, either on the reference spectrum itself or in the information that accompanies it, the manufacturer's or supplier's company name and lot number, the date the entry was generated, and his or her initials; or

2. The spectral information in the entry must be matched to information for the same compound that is published in an approved library or literature. The person that performs the comparison must note, either on the reference spectrum itself or in 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.

Preparer

Ruben A. Rendon, Jr. Date: 11/21/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 11/21/2008
Quality Assurance

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	09/01/2001	Original Issue
	05/15/2002	Numbering 2.1, 1 Delete evidence destruction move to LOG 5.2 and evidence handling to 2.2 of this chapter, 2.1, 4 Added Library References 2.2 Delete evidence destruction citation found in LOG 5.2
01	12/01/2002	Minor Revision, Text from chapter 2 Modification Section 4, 1 "…the spectra will be compared to a reference standard. The source of the spectrum of this standard will be documented in the case file". Modification Section 4, 2 "…checklist, or complete listing of references will be used to denote the common references for standard spectra."
02	07/01/2003	Minor revision with respect to title and reference to IR changed to FTIR
03	12/01/2003	Addition Section 4 B "References used for Pharmaceutical ID will be documented in case file." Modification to Section 4 C "The Approved List of Reference Abbreviations (Appendix A) will be used…."
04	05/01/2005	Addition Section 1 from LOG-03-08 v.01
05	07/20/2007	Addition of Section 6 Reference Spectral Libraries
06	02/09/2009	Major revision – Section 4 Advisory Board 11/20/2008

Appendix A—Approved List of Reference Abbreviations

Abbreviation	Resource
CND	CND Analytical
G	Georgia Bureau of Investigation (Georgia State Crime Lab) Library
J	Journal (Microgram, CLIC, Journal of Forensic Science, etc.) *
L	In-house Laboratory Library
M	Mills, Roberson Instrumental Data for Drug Analysis
N	NIST (NBS) Library
A	American Academy of Forensic Science drug library
C	Clarke's Isolation and Identification of Drugs
IC	Mattson ICON Library
PMW	Pfleger/Mauer/Weber drug Library
AL	Aldrich Library of IR Spectra
FL	Fluka IR Library
PDR	Physicians Desk Reference
LOGO	DEA Logo Index
ID	Ident-a-drug
RX	Rx List, http:\\www.rxlist.com
RXID	Amer-Chem Rx-ID
DEF	Diccionario de Especialidades Farmacéuticas
PC	Poison Control
DIB	Drug ID Bible

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

Note: If the reference used has several volumes/versions, then you 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.

Preparer

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Controlled Substance Advisory Board Chair

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Zoe M. Smith Date: 11/21/2008
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	05/15/2002	Original Issue
01	12/01/2002	Minor Revision
02	12/01/2003	Title change Addition of approved references
03	05/01/2005	Modification of abbreviations: CND, CND Analytical C, Clarke's Isolation and Identification of Drugs DIB, Drug ID Bible
04	10/10/2006	Modification of abbreviations/reference for DEF
05	02/09/2009	Major revision Advisory Board 11/20/2008

INSTRUMENT PERFORMANCE AND MAINTENANCE

1 Scope

The policy regarding instrument performance and maintenance for Controlled Substances is established in these quality assurance guidelines.

2 Maintenance and Performance of Laboratory Instrumentation

2.1 General Requirements for Analytical Instrumentation

A. All instruments' performance will be verified.

B. All instruments' performance will be re-verified if they are moved or if a major repair is performed.

C. If an instrument fails calibration or a performance verification check, or if a performance problem is detected during casework, the instrument must be removed from service. The supervisor must be notified and the problem recorded in the logbook.

D. No instrument is to be used if it is not in proper working order.

E. Repair or have the instrument repaired and perform routine quality control procedures to ensure it is working properly before the instrument is returned to service.

F. The supervisor will determine if the instrument is ready to return to service for routine casework.

G. Each laboratory must develop and implement an annual maintenance plan for the following instruments: GC, LC, GC/MS, UV, and FTIR.

H. Keep a record of all repairs and the routine maintenance in a logbook.

2.2 UV/VIS Spectrophotometer

1. Conduct a performance verification check on UV/VIS instruments at least once each quarter of the calendar year.

2. 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 condition of the UV/VIS instrument.

3. 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.

4. Scan the standard following the manufacturer's recommendations and record the results. Compare the peak wavelengths given by the manufacturer of the standard to the measured wavelengths, and either mark the results on the spectrum or list them in tabular form.

5. If the value is not within acceptable limits, corrective maintenance must be performed before the instrument can be used for casework.

6. Maintain a logbook with the results.

2.3 FTIR Spectrophotometer

1. Conduct a performance verification check on FTIR instruments at least once each quarter of the calendar year.

2. Follow the manufacturer's instructions for performance verification.

3. Scan and record the infrared spectrum of a polystyrene film standard, in percent transmittance versus the wave number, and examine the peaks at 3060, 1601, 1583, and 1028 cm^{-1}. The peaks should be within 4 cm^{-1} of their expected values to pass the verification test.

4. Maintain a logbook with the results.

2.4 Gas Chromatography/ Mass Spectrometry (GC/MS)

A. Performance Verification Check

1. The detector must be tuned at least monthly or more often as needed.

2. The instrument must be tuned according to established criteria for a successful tune. It is recommended that specifications used to check the instrument performance be kept with the logbook.

3. If the GC component is used for retention time or quantitation, a known drug reference standard must be analyzed to verify acceptable instrument performance for the instrument to be used for analysis of evidentiary materials.

4. Maintain a logbook with the results.

B. Other GC/MS maintenance

1. Run a solvent blank at least quarterly or more often if needed. A record for each such blank shall be maintained.

2. Each lab shall have a preventative maintenance schedule for the major components.

3. A logbook documenting all maintenance will be kept with the instrument.

2.5 Gas Chromatograph (GC)

A. Performance Verification Check

1. A known drug reference standard must be analyzed to verify acceptable instrument performance for the instrument to be used for analysis of evidentiary materials.

2. Maintain a logbook with the results.

B. Other GC maintenance

1. Each lab shall have a preventative maintenance schedule.
2. A logbook documenting all maintenance will be kept with the instrument.

2.6 Balances

1. Each quarter of the calendar year, laboratory personnel will verify balance accuracy, using standard weights. Balances will be verified whenever they are moved from one location to another. Laboratory standard weights should be verified after the annual balance re-certification.
2. Balances should be certified by an external vendor at least once a year.
3. Keep the balances clean at all times and check the level frequently.
4. The appropriate balance will be used for weight being measured and precision required. Care should be taken to not overload a balance.
5. Since the tolerances of balances vary, the instrument specifications must be checked to determine the appropriate criteria for satisfactory performance.
6. Maintain a log book with the results of the balance checks, maintenance, and certification.
7. Each lab shall establish performance criteria specific to their balances.

3 Record

Logbook or equivalent documentation

Preparer

Ruben A. Rendon, Jr. Date: 11/21/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 01/22/2009
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 2 "Calibration" replaced with "performance verification check" where applicable. Deletion Section 2.1, 4 "Peak locations should be within 2 nm." Modification Section 2.3, A, 1 "The Mass Selective Detector (MSD) must be tuned at least monthly or more often as needed. " Modification Section 2.3, A, 2 a "…mass-to-charge ratios (m/z) are assigned correctly and that the scan ratio is set properly. The procedure also serves as a check for air leaks." Deletion Section 5.4, A, 2, b-e Addition Section 2.3, A, 2, b "The instrument must be tuned according to the manufacturer's instructions and must meet the manufacturer's specifications." Addition Section 2.3, A, 2, c "Maintain a logbook with the results." Modification Section 2.3 B, 1 "Run solvent blank as often as needed. A record for each such blank should be maintained."
01	07/01/2003	Modification Section 2.1 reference to spectrophotometer changed to UV/VIS instruments Modification Section 2.2 reference infrared spectroscopy changed to FTIR spectrophotometry and infrared spectrophotometer to FTIR instruments Deletion Section 2.2 4 with respect to lab establishing performance criteria Deletion Section 2.3 A. 2 Minor numbering

Version #	Effective Date	Brief Description of Change(s)
02	10/10/2006	Modification to Section 1 "The policy regarding instrument performance and maintenance for Controlled Substances is established in these ~~to establish~~ quality assurance guidelines ~~for the maintenance, performance, and repair of analytical instrumentation and balances~~." Minor Revision Section 2.1 rearrangement of text from Section 2.5. Modification Section 2.2 #2 "A ~~Holmium Oxide filter~~ standard consisting of either holmium oxide glass or a solution of holmium oxide in sulfuric or perchloric acid ~~may~~ shall be used to verify the condition of the UV/VIS instrument. ~~Alternatively, a cell using benzene vapor may be used to verify the performance of the UV/VIS instrument.~~" Modification Section 2.2 #3-6 regarding performance specifications for UV/VIS. Modification to Section 2.3 #3 "Scan and record the infrared spectrum of a polystyrene film standard, ~~provided by the instrument manufacturer,~~ in percent transmittance versus the wave number, and examine the peaks at ~~3025~~3060, ~~1600~~1601, 1583, and 1028 ~~and 698~~cm^{-1}…." Modification to Section 2.4 A #2 "The instrument must be tuned according to ~~the manufacturer's instructions and must meet the manufacturer's specifications~~ established criteria for a successful tune." Addition to Section 2.4 A #3 "It is recommended that the manufacturer's specifications used to check the instrument performance be kept with the logbook." Modification to Section 2.4 B #2 "Each lab shall have a preventative maintenance schedule for the major components. ~~Perform regular and preventative maintenance according to the manufacturer's recommendations~~." Modification to Section 2.4 B #3 "A logbook documenting all ~~non-routine~~ maintenance ~~(e.g. column replacement, filament replacement, seal replacement, vacuum oil changes, source cleaning, and major repairs)~~ will be kept with the instrument." Deletion Section 2.5 moved and modified to Section 2.1. Modification to Section 2.5 #1 "…will ~~check~~ verify balance accuracy, using standard weights. Balances will be ~~checked~~ verified whenever they are moved from one location to another. Laboratory standard weights should be ~~checked~~ verified after the annual balance re-certification ~~of the balance~~." Modification to Section 2.5 #2 "Balances must ~~should~~ be certified…." Deletion Section 2.5 #6 regarding general balance specifications table. Modification to Section 2.5 #7 "~~It is recommended that~~ Each lab shall establish performance criteria specific to their balances ~~and bulk scales~~."

Version #	Effective Date	Brief Description of Change(s)
03	07/20/2007	Modification Section 2.4, B, 1: Run a solvent blank ~~as often as needed~~at least quarterly or more often if needed. A record for each such blank ~~should~~ shall be maintained.
04	05/05/2008	Major revision – Sections 2.4 and 2.5 Advisory Board 02/27/2008
05	02/09/2009	Major revision – Section 2.2 Advisory Board 11/20/2008

REAGENTS

1 Scope

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

2 Practice

A. All reagents may be prepared in any desired volume so long as the proportions specified in the individual instructions are maintained.

B. For prepared reagents (including simple dilutions) that are not quality checked, a reagent preparation logbook will be maintained that includes the following information:

1. Name of the solution
2. Reagent preparation date
3. Initials of the person who prepared the reagent

C. For each reagent that requires performance verification prior to their use in casework, a reagent quality control logbook or equivalent notation in the case record will be maintained that includes the following information:

1. Name of the solution
2. Reagent preparation date
3. Initials of the person who prepared the reagent
4. Date the reagent was quality tested
5. Reagent performance result
6. Initials of the person who quality tested the reagent

D. The following reagents and/or reagent systems are subject to performance verification:

1. General reagents

a) *Cobalt Nitrate Reagent*

b) *Cobalt Thiocyanate Reagent*

c) *p-Dimethylaminobenzaldehyde Reagent*

d) *Duquenois-Levine System*

e) *Fast Blue B Reagent*

f) *Formaldehyde/Sulfuric Acid Reagent*

g) *Gold Bromide Reagent*

h) *Gold Chloride Reagent*

i) *Liebermann Reagent*

j) *Marquis Reagent*

k) *Mercuric Iodide Reagent*

l) *Modified Schweppe's Reagent*

m) *Silver/Copper Nitrate Reagent*

n) *Sodium Nitroprusside System*

2. TLC indicator reagents (verified when performing Thin-Layer Chromatography, CS-06-01)

a) *Acidified Iodoplatinate Reagent*

b) *Fast Blue RR Reagent*

c) *Ninhydrin Reagent*

d) *Potassium Permanganate reagent*

E. Reagents subject to performance verification which are stored will have their performance verified monthly, unless

a) *the individual reagent's instructions specify a shorter interval or*

b) *the reagent has not been used for a month or more.*

F. No reagent or other chemical preparation will be used in casework if it is not working properly or if it is contaminated.

G. Examiners who suspect that a reagent, chemical preparation or solvent is not working properly or is contaminated must:

1. Check the reagent or system with proper control samples.
2. Discard the reagent if it fails the quality check, prepare new reagent, and quality check the reagent with a known standard.
3. Refrain from using the reagent in laboratory case work until the problem has been corrected. Inform the quality manager if the problem persists.

3 Record

Log books or equivalent documentation

Preparer

Larry Todsen Date: 10/06/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 10/06/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 2 Modification Section 2 A "…following reagents or reagent systems:" Addition Section(s) 2 A h, I, k-s
01	07/01/2003	Modification Section 2 B 3 "Reagent Performance result" and deletion periodic documented checks Addition Section 2 C frequency of testing "Reagent performance will be verified monthly and the results of the quality check documented, unless otherwise specified in the procedure. If the reagent has not been used for a month or more, it must be checked and documented with a standard before it may be used on case samples." Minor revision Section 2 D Minor revision Section 2 E
02	12/01/2003	Addition Section 2 A (t) "Modified Schweppe's Reagent "
03	05/01/2005	Deletion Section 2 A (f) "Marihuana Thin Layer System" Modification to Section 2 A (c) "~~Cocaine System~~ Cobalt Thiocyanate Reagent(s)"
04	10/10/2006	Major revision; reorganization and clarification of text.

EXAMINATION OF CONTROLLED SUBSTANCES, DANGEROUS DRUGS AND RELATED COMPOUNDS

1 Scope

To describe a basic analytical scheme, utilizing screening tests, extraction techniques, and instrumental analytical procedures, for the isolation and identification of controlled substances, dangerous drugs and other compounds such as clandestine laboratory reagents and precursors.

2 Safety

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

3 Related Documents

Evidence Record Sheet (Lab-22)

Laboratory Submission Form (Lab-6)

Controlled Substance Worksheet (LAB-CS-01)

Controlled Substance Worksheet Instructions (CS-03-01A)

Marihuana Worksheet (LAB-CS-02, optional)

Examination and Destruction of Excess Quantity Controlled Substances (CS-03-02)

4 Procedure

Document the observations and examinations on the approved controlled substances worksheets (LAB-CS-01 or LAB-CS-02). Other supplemental documents may be used.

4.1 Retrieval and Initial Examination of the Evidence

1. Retrieve the evidence following laboratory policy concerning barcodes and the transfer of the items to the analyst.
2. Compare the information on the *Evidence Record Sheet* and the *Laboratory Submission Form* to the physical evidence and make sure all information is consistent regarding the description of the evidence, condition of seals and case number. If any changes have occurred, such as leakage of the contents, make a note on the worksheet.

3. If there is reason to believe that specific types of evidence may be found on particular items, other examiners may be requested to assist in the description, collection, and analysis of that material (i.e. hair/fiber, blood, body fluids, residue, etc.) and care should be taken to preserve trace evidence for analysis by other sections. Examine the evidence, preferably with gloved hands.

4. Mark each exhibit of evidence clearly with the laboratory case number and analyst's initials. Small items may need to be repackaged in order to place identifying marks upon them.

5. Write a brief description of each item.

6. For tablets and capsules, record the physical description, including any significant markings and logos. These markings and logos may be compared with pharmaceutical reference literature. Record the reference(s) used for comparison.

7. The use of abbreviations is acceptable as long as they are obvious or a key is available.

8. Record the number of individual items received. Large numbers of tablets or capsules may be approximated by using weight calculations.

9. Record any repackaging done by the analyst or any significant changes to the exhibit.

10. Separate (sub-divide) exhibits with multiple items if any of these items appear to be different. Analyze as separate exhibits.

11. If individual items and/or containers are not numbered or are numbered incorrectly, the numbering should be resolved. When necessary, the analyst may contact the submitting officer for clarification. Documentation of corrective action must be included in the case folder.

12. If plants are submitted, they should be air dried in a secure area of the laboratory where possible.

13. Care must be exercised to avoid cross-contamination if more than one item of evidence is open at the same time.

14. If the entire evidence exhibit and/or sub-evidence exhibit is consumed during its analysis:

    a) *a remaining analytical sample will need to be retained as evidence if determined to be positive; and*

    b) *document analytical sample disposition.*

4.2 Procedure for Weighing Samples

1. Use the appropriate balance for the amount of sample to be weighed.

2. Clean the balance pan (if necessary) and zero the balance prior to weighing the sample.

3. Record the tare weights when used. If the source of the tare weight is not obvious, make notes describing how the tare was calculated. Also, make appropriate notes if an estimated tare weight is used.

4. If before analysis and tare weights are not in the same units, the tare must be converted for determination of the net weight:

   a) *Conversion factor used should be 28.4 g/oz or 454.4 g/lb.*

   b) *Convert tare weight to same units as the before analysis weight; do not truncate values*

   c) *Subtract tare weight from before analysis weight to determine net weight*

5. Record the net weight of the sample, excluding the weight of any packaging.

   a) *If cigarettes or cigarette butts require analysis and the net weight is critical for determining the penalty group, separate the plant matter from the cigarette paper to determine the net weight.*

   b) *If whole marihuana plants are submitted, remove the mature stalk and roots if present and record the net weight of the remaining plant material.*

   c) *If a net weight cannot be determined, an appropriate footnote will be used.*

   d) *If the net weight is less than 0.01 gram, it may be recorded as trace.*

6. Document all weights, and information used to calculate weight (excluding weight of any packaging). The volume of liquid samples may be recorded.

7. The analyst should determine the tare weight in a manner appropriate for that case. The method used will be documented on the worksheet. Where it is impractical or unnecessary to determine a tare weight, the analyst may report the gross weight with an appropriate footnote.

4.3 Evidence Sampling Techniques

A. Multiple items (other than commercially-prepared tablets and capsules)

1. When multiple items of a suspected controlled substance are submitted as one exhibit to the laboratory for analysis, the analyst must use discretion and sample the number of items for testing that is sufficient for that particular case. At a minimum:

   a) *a sufficient number of items must be selected to attain the maximum applicable weight limit as delineated in the Texas Controlled Substances Act and initially must be subjected to at least preliminary testing; or*

   b) *in accordance with the sampling plan given below, a number of items must be randomly selected, and the samples initially must be subjected at least to preliminary testing. This sampling plan can be used to prove that a statistically significant percentage (90%) of the total population will be positive with respect to the testing performed, with 95% certainty.*

Total Population	Required Number of Consecutive Positives
5	All (4 gives 80% certainty)
10	All (9 gives 90% certainty)
20	16
30	19
40	21
50	22
75	23
100	25
250	27
500	28
1000+	29

2. Except for suspected marihuana, if the individual samples selected from a multi-item exhibit are similar in physical appearance and yield the same results from presumptive testing, then a composite or composites of those samples may be used for confirmatory instrumental analysis.

3. For suspected marihuana, each sample that is selected from a multi-item exhibit must be examined microscopically. If all the samples yield a positive microscopic result, presumptive testing may be performed either on each of the selected samples or on composites of the selected samples.

4. Regardless of the sampling technique used, if one or more negative samples are found mixed with items containing a controlled substance, or if preliminary testing does not give consistent results, then each item in the exhibit must be analyzed separately.

B. Liquid Samples

1. Take care to ensure that a homogeneous sample is collected for analysis.

2. If two layers are present, separate into two exhibits and analyze each layer as a separate sample.

C. Powdered (Solid) samples

1. Take care to ensure that a homogeneous sample is collected for analysis.

2. If more than one color or type of substance is present, either homogenize the sample by grinding in a clean mortar with a clean pestle or physically separate the particles to be analyzed separately.

D. Tablets and capsules

1. If the tablets or capsules appear to be clandestine or counterfeit in origin, or if they appear to have been altered, follow the sampling plan for analysis.

2. If the tablets and capsules appear to be commercially-prepared pharmaceuticals and there is no reason to suspect tampering or counterfeiting, then one tablet or capsule may be analyzed.

4.4 Basic Analytical Scheme

The basic analytical scheme for the analysis of suspected controlled substances, dangerous drugs and other related compounds consists of sample preparation and extraction or isolation procedures in various combinations with the following tests and instrumentation. Reference specific analytical tests and special preparatory or extraction procedures used that are located elsewhere.

The analyst must determine the appropriate sampling techniques, methods of recovery, extraction procedures and instrumental analysis to be used for identification of a compound on a case-by-case basis.

A. Required Analytical Procedures

1. Number of tests that are required

   a) One confirmatory instrumental test (either FTIR or GC/MS) and at least one different positive test (including TLC, GC, GC/MS, FTIR, UV/VIS, Microcrystalline, preliminary pharmaceutical examination, or Presumptive [Color/Odor] Test) is required for identification of an unknown substance.

   b) Microscopic identification is required, and at least one other positive test (Duquenois-Levine, TLC, GC, GC/MS, or FTIR) is required to indicate the presence of THC and/or Cannabinoids in marihuana samples, excluding seeds.

   c) 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.

2. Each test will be conducted from an independent sample when possible. In cases with a limited sample, more than one test may be performed using the same sample.

3. For exhibits that contain an insufficient amount of material for two independent samples or 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 before the evidence sample (e.g. when packaging is rinsed to sample the contents of an exhibit). Blanks can be prepared and analyzed, if desired, for other instrumental techniques as well.

4. 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 at the discretion of the analyst.

5. Each test will be documented.

6. Quantitate all solids that weigh two (2) kilograms or more and liquids with a volume of four (4) Liters or more containing Cocaine, Methamphetamine, or Heroin. An exception will be exhibits from clandestine laboratory investigations which are in concentrations less than 1%. The procedures used to analyze and report such exhibits must be documented.

7. When an identified controlled substance is listed in two or more penalty groups, quantitation may be necessary to facilitate the establishment of the appropriate penalty group for that exhibit.

B. Sample Preparation and Basic Extraction Procedures

1. Determined by the type and amount of sample present, as well as analytical technique or instrumental analysis to be performed.

2. Document the sample preparation and extraction technique used to isolate the compound of interest.

3. Neat samples are undiluted samples that require little or no preparation prior to analysis.

4. Incorporated in a KBr pellet for Infrared spectrophotometry

   a) *Usually solids*

   b) *Useful for some pharmaceuticals (such as antibiotics), some vitamins, and some dilutants*

5. Direct in a vapor cell or as a thin film between salt cells for infrared spectrophotometry of liquid samples

6. Dilute with a suitable solvent for GC/MS or use headspace analysis procedures

7. “Dry” extractions with organic solvents

8. The extraction solvent used to prepare marihuana samples will be petroleum ether unless documented in the case file.

9. Organic solvent / activated base

10. Partitioning between aqueous and organic phases

    a) *Aqueous acid / organic solvent extractions*

    b) *Aqueous base / organic solvent extractions*

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 file.*

11. Column chromatography

12. Distillation procedures

13. Micro-diffusion cleanup procedure

14. Preparatory thin layer chromatography

15. Derivative analysis, including:

    a) *Acetic anhydride, benzoyl chloride or phenyl isothiocyanate (PIT)*

    Amphetamine and methamphetamine

    MDA derivatives

    b) *Bis(trimethylsilyl)trifluoroacetamide (BSTFA)*

    GHB

    LSD

    Psilocybin from mushrooms

16. It should be noted that specific extraction schemes given in these Standard Operating Procedures are examples of commonly accepted procedures. However, there are numerous methods described within the training manual which have been validated and are equally acceptable.

C. Proceed to the appropriate approved analytical tests

4.5 Concluding Examination and Return of the Evidence

1. Record an after analysis weight for all reported substances except for marihuana, other plant material, and cases greater than one kilogram. If the after analysis weight is less than 0.01 grams, ***"trace"*** should be indicated. Make a note if the entire sample is consumed in analysis.

2. Document the number of tablets or capsules used in analysis, the number remaining, or the after analysis weight.

3. The gross inventory weight will be recorded for DPS retained cases of five pounds or more.

4. All original exhibits should be re-packaged in the original container if possible. The evidence should be re-sealed in a manner that would detect tampering. If samples are taken from an exhibit for preservation, the samples will be packaged appropriately and sealed in a manner that would detect tampering.

5. The evidence should be transferred to the evidence custodian or filed in the laboratory vault until its final disposition.

5 Literature and Supporting Documentation

Ginn, Bill; "Statistical Validity of Random Sampling", in-house DPS publication, April, 1992.

R. S. Frank, S. W. Hinkley, and C. G. Hoffman, "Representative Sampling of Drug Seizures in Multiple Containers," *Journal of Forensic Sciences*, 36 (1991): 350-357.

Preparer

Ruben A. Rendon, Jr. Date: 03/03/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 04/08/2008
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
	05/15/2002	Section 4.2 (4) Added information for calculation of net weight when units are not the same. Section 4.8 Added information on destruction and examination of excess quantity.
01	12/01/2002	Minor Revision Addition Section 4.4, A, 1 (b) "…indicate the presence of THC and/or Cannabinoids in marihuana samples." Modification Section 4.4, A 6 "Quantitate all solids weighing 200 grams or more and liquids with a volume of 1 liter or more which contain penalty group 1 or 2 substances. An exception would be exhibits from clandestine laboratory investigations which contain penalty group 1 or 2 substances in concentrations less than 0.1%. The procedures each laboratory will use to analyze and report such exhibits must be documented." Deletion from Section 4.5 2 not required to record the after-analysis weight of tablets or capsules. Examination and Destruction of Excess Quantity of Controlled Substances moved to separate document.
02	07/01/2003	Minor revision with respect to title and reference to IR changed to FTIR Addition Section 3.1 4 h "If plants are submitted, they should be air dried in a secure area of the laboratory where possible." Addition Section 3.2 5 a-c a. "If cigarettes or cigarette butts require analysis and the net weight is critical for determining the penalty group, separate the plant matter from the cigarette paper to determine the net weight." b. "If whole marihuana plants are submitted, remove the mature stalk

Version #	Effective Date	Brief Description of Change(s)
02	07/01/2003	and roots if present and record the net weight of the remaining plant material." c. "If a net weight cannot be determined, an appropriate footnote will be used." Modification Section 3.4 A. 1 (a) references to IR changed to "FTIR", deletion "controlled" Modification Section 3.4 A. 1 (b) reference to IR changed to "FTIR", addition "excluding seeds" Deletion Section 3.4 A 2 moved to CS0101 Modification Section 3.4 A 2 "For exhibits that contain an insufficient amount of sample for two independent tests, a method blank must be prepared using the same parameters as the evidence sample and analyzed before evidence sample (i.e. when packaging is rinsed to sample the contents of an exhibit). " Modification Section 3.4 A 5 "Quantitate all solids that weigh 400 grams or more and liquids with a volume of 1 liter or more containing Cocaine, Amphetamine, Methamphetamine, or Heroin. An exception will be exhibits from clandestine laboratory investigations which are in concentrations less than 1%. The procedures used to analyze and report such exhibits must be documented." Addition Section 3.4 A 6 "When an identified controlled substance is listed in two or more penalty groups, it is necessary to facilitate the establishment of the appropriate penalty group for that exhibit." Modification Section 3.4 C changed entire section to "Proceed to appropriate approved analytical tests" Modification Section 3.5 "Record a net *after analysis* weight for all reported controlled substances except for tablets and capsules, marihuana, and other plant material. If the remaining powder sample is less than 0.01 grams, ***"trace"*** should be indicated as the after analysis amount. Make a note if the entire sample is consumed in analysis. For marihuana and other plant material: It is not required to record net after analysis weight or amount consumed in analysis. For tablets or capsules: Document the number used in analysis, the number remaining, or the net after analysis weight. Empty capsules should be retained with the evidence."
03	12/01/2003	Addition Section 3.1 #4 (c) "If Pharmaceutical ID is used as a test, record the reference used. " Modification to Section 3.1 #4 (g) "…appear to be different in content."

Version #	Effective Date	Brief Description of Change(s)
03	12/01/2003	Addition Section 3.2 #5 (d) "If the net weight is less than 0.01 gram, it may be recorded as trace." Modification to Section 3.4 A #1 (a) "…and at least one <u>different</u> positive test…." Addition Section 3.4 A #1 (c) "Dangerous drugs may be identified by Pharmaceutical ID only when no analytical data is available (reference or standard)." Modification to Section 3.4 A #2 "Each test <u>will</u> be conducted from an independent sample when possible. In cases with a limited sample, more than one test may be performed using the same sample. " 1. Addition Section 3.4 A #4 "If positive results can only be obtained from GC/MS, then appropriate standards may be run to use the retention time as the second positive test." Modification to Section 3.5 #1 "Record an after analysis weight for all reported substances except for marihuana, <u>bulk cases greater than one kilogram</u>, and other plant material. If the <u>after analysis weight</u> is less than 0.01 grams, ***"trace"*** should be indicated. Make a note if the entire sample is consumed in analysis." Modification to Section 3.5 #2 "Document the number <u>of tablets or capsules</u> used in analysis, the number remaining, or the after analysis weight." Addition Section 3.5 #3 "The gross inventory weight will be recorded for DPS retained bulk cases of five pounds or more."
04	11/01/2004	Addition Section 3.1 #3 allowing for involvement of other examiners from other disciplines on the examination Modification to Section 3.1 #5 g "…appear to be different~~ in content~~…." Modification to Section 3.2 #7 "~~When weighing bulk cases,~~ The analyst should determine the tare weight in a manner appropriate for that case. The method used will be documented on the worksheet. ~~In bulk cases,~~ Where it is impractical or unnecessary to determine…." Modification to Section 3.3 A. #1 requiring "…<u>At a minimum, either a sufficient number of packages must be analyzed that surpasses</u> ~~This may include analyzing enough packages to surpass~~ a particular weight limit as listed in the Texas Health and Safety Code §481 or <u>the selection of a random number of samples as defined in the statistical sampling plan…."</u> Modification to Section 3.5 #1 "…except for marihuana, <u>other plant material, and</u> ~~bulk~~ cases greater than one kilogram~~, and other plant~~

Version #	Effective Date	Brief Description of Change(s)
04	11/01/2004	~~material~~….." Modification to Section 3.5 #3 "…DPS retained ~~bulk~~ cases…." Modification to Section 3.5 #4 "…exhibit for preservation~~, such as in bulk cases~~…."
05	05/01/2005	Minor Revision Section 3.3 A #1 for clarification Minor Revision with respect to Controlled Substances Worksheet replacing Lab c-16 with LAB-CS-01
06	10/10/2006	Addition Section 3 Related Documents Addition to Section 4 "Document the observations and examinations on the approved controlled substances worksheets (LAB-CS-01 or LAB-CS-02). Other supplemental documents may be used." Deletion Section 5 Records Modification to Section4.1 #5 "Write a brief description of each item ~~in the Description column."~~ Modification to Section 4.1 #6 "For tablets and capsules, record the physical description, including any significant markings and logos. These markings and logos may be compared with pharmaceutical reference literature. ~~If Pharmaceutical ID is used as a test,~~ Record the reference(s) used for comparison." Modification to Section 4.3 A #1 "Multiple ~~container~~ items", "When multiple ~~containers~~ items of a suspected controlled substance…analyst must use discretion and perform analyses on the number of ~~packages~~ items that is sufficient for that particular case. ~~Either~~ At a minimum, a sufficient number of ~~packages~~ items must be analyzed to attain … a number of random ~~number of samples~~ items must be selected …." Modification to Section 4.3 B #2 "If two layers are present, ~~either~~ separate into two exhibits and analyze each layer as a separate sample~~ or take proportional samples from each for a combined analysis~~." Modification to Section 4.3 C #2 "…physically separate the particles~~, using forceps or another method to collect portions of each type~~ to be analyzed separately." Modification to Section 4.3 D #1 "If the tablets or capsules appear to be clandestine or counterfeit in origin, or if they appear to have been altered, ~~randomly select a number of tablets~~ follow the sampling plan for analysis"

Version #	Effective Date	Brief Description of Change(s)
06	10/10/2006	Modification to Section 4.4 A #1 (a) "…preliminary pharmaceutical examination ~~ID~~…" Deletion Section 4.4 A #1 (c) "~~Dangerous drugs may be identified by pharmaceutical ID only when no analytical data is available (reference or standard).~~" Addition Section 4.4 A #1 (c) "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." Modification to Section 4.4 A #6 "Quantitate all solids that weigh ~~400 grams~~ two (2) kilograms or more and liquids with a volume of ~~1 Liter~~ four (4) Liters or more containing Cocaine, Amphetamine, Methamphetamine, or Heroin." Deletion Section 5 regarding record
07	07/20/2007	Modification Section 4.3, 1: regarding evidence sampling techniques of multiple items Addition Section 4.4, A: 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 at the discretion of the analyst. Addition of Section 4.4, B, 8: The extraction solvent used to prepare marihuana samples will be petroleum ether unless documented in the case file. Modification Section 4.4, B, 10: Partitioning between aqueous and organic phases ~~solvent extraction~~ Aqueous acid / organic solvent extractions Aqueous base / organic solvent extractions *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 file.*
08	05/05/2008	Major revision – Sections 4.1 and 4.4 Advisory Board 02/27/2008

INSTRUCTIONS FOR CONTROLLED SUBSTANCES WORKSHEET, LAB-CS-01

1 General Instructions

Please print or type all information on the form. The form is available electronically to facilitate completion on a computer.

The information requested on this form is needed to document the examination of substances.

2 Case Information

Lab Case Number (*Required*) –this is your cross-reference to the laboratory report.

Analyst (*Required*) – Initials of the person who analyzes the case.

Date Started (*Required*) – The date evidence is opened for analysis.

Date Completed (*Required*) – The date laboratory examinations are completed.

Page – *Required*, when more than one worksheet is used.
Gross Inventory Wt – *Required*, when the gross inventory weight is five (5) pounds or more.

Complete the *Outside container* box (*Required*) and the *Inside container* box (*Optional*) with the appropriate description of the evidence container.

Additional Notes (*Optional*)

3 Examination Information

3.1 Evidence Description (*Required*)

Exhibit # - The exhibit number as listed on the laboratory submission form. 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.

Items – number of individual items received.

Analyzed – number of individual items on which examinations were performed.

Exams – total number of examinations performed.

Description of Evidence – Although there is not a specific heading for description of evidence, the space below the listing of the exhibit # will be used for brief descriptions of each item of evidence.

3.2 Weights

Balance ID (*Required, if a weight is determined*) – The ID of the balance that was used to weigh the substances

Before Analysis (*Optional*) – the gross weight of the exhibit

Tare (*Optional*) – weight of packaging

Net (*Required, if a weight is reported*) – this is the net weight of the substance that shall be reported

After Analysis – If required, weight or number of items of evidence remaining after analysis. (Refer to CS-03-01 section 3.5.)

3.3 Preliminary Examinations

Record observations such as +, -, color, number of tests performed, etc. as appropriate for the respective test(s).

A. Listed Possible Preliminary Examinations

1. Marquis
2. SNP
3. $Co(SCN)_2$
4. Duquenois
5. TLC

B. For other (non-listed) tests, list the tests that were conducted and their respective results

3.4 Instrumental Examinations

Record observations such as √, +, -, number of tests performed, reference used, etc. as appropriate for the respective test(s).

A. Listed Possible Instrumental Examinations

1. Microscopic
2. FTIR
3. GC/MS
4. UV Acid
5. UV Base

B. For other (non-listed) tests, list the tests that were conducted and their respective results

3.5 Conclusions

Substance Identified (*Required*) – the results of the analysis

Class (*Optional*) – a number used in DRAGNet to classify drug results for statistical purposes

Preparer

Larry Todsen Date: 06/12/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 06/12/2006
Quality Assurance Specialist

Version #	Effective Date	Brief Description of Change(s)
00	05/01/2005	Original Issue
01	10/10/2006	Modification to Section 2 "Page – *Required*, when more than one worksheet is used. Complete the *Outside container* box (*Required*) and ~~(if applicable)~~ the *Inside container* box (*Optional*) with the appropriate description of the evidence container." Modification to Section 3.3 B "…non-listed tests, ~~should~~ list the test…." Modification to Section 3.4 B "…non-listed tests, ~~should~~ list the test…."

EXAMINATION AND DESTRUCTION OF EXCESS QUANTITY OF CONTROLLED SUBSTANCES

1 Scope

Provisions for examination and destruction of evidence 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 §481.160 and Texas DPS Administrative Code Title 37 Part 1.

2 Definition

2.1 Excess quantity is defined as greater than:

A. 50 pounds of marihuana

B. two (2) kilograms of dry evidence, such as powder

C. 500 milliliters of liquid precursor or controlled substances

D. 200 dosage units of an item such as tablets, capsules, and liquids

E. Five individual controlled substance plants, such as marihuana or peyote

2.2 Modifications to excess quantity definition

Any modifications to the defined amounts of excess quantity by laboratory policy must have express approval of the appropriate prosecuting authority under the provisions of the Texas DPS Administrative Code Title 37 Part 1 §13.157(d).

3 Practice

3.1 Weighing/Examinations

A. All evidence packages must be either marked with case number upon receipt or segregated and sampled within five business days of receipt.

B. Conduct examinations as required under Examination of Controlled Substances, Dangerous Drugs and Related Compounds, CS-03-01.

C. The gross inventory weight of the evidence retained must be determined and recorded on the evidence worksheet. This weight includes the weight of the samples retained plus the weight of the boxes or containers.

3.2 Photographing

A. At least one representative photograph, image or video recording must be taken and preserved that is sufficient to reasonably depict the total amount of evidence.

B. Photographs, digital media and/or video that are representative of excess quantity controlled substances will be maintained as evidence.

C. Duplicate copies of evidentiary photographs may be prepared for the submitting agency and/or as documentation photographs.

3.3 Marking for Identification

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

B. All retained samples and bundles may be combined into a convenient container.

3.4 Retained Samples

A. A minimum of five random and representative samples must be collected from the total amount of property or plant in the case and a sufficient quantity, which is at least that amount defined as excess quantity, preserved to provide for discovery.

B. One representative liquid sample must be retained, which is at least that amount defined as excess quantity. In the case of illicit chemical laboratories as defined under §481.160 section (d) of the Health and Safety Code, a sample of reduced size may be retained if it is determined to be material that is too hazardous to handle or safely store.

C. At least one complete package unit should be retained (e.g. one brick, bale, bundle, box, etc.).

D. The retained samples and bundles must be stored in a secure location until the disposition of the case.

4 Destruction of the Excess Quantity

A. After analysis, the laboratory may proceed with destruction of excess quantity according to Destruction of Evidence (LOG-05-02), with the written authorization from the submitting peace officer, the submitting law enforcement agency, or the office of the prosecutor responsible for the case in accordance with the provisions of Texas Health and Safety Code §481.160 and Texas DPS Administrative Code Title 37 Part 1 §13.157(e).

B. If the laboratory has a blanket written authorization from the submitting law enforcement agency or the office of the prosecutor, then the excess quantity may be destroyed after analysis.

C. 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.

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

E. All excess quantity will be destroyed after analysis, unless prior arrangements have been made to store it.

5 Record

Case File

Preparer

Ruben A. Rendon, Jr. Date: 11/21/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 01/22/2008
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue; Minor Revision; Text from Chapter 3 Text moved to LOG requiring logging and marking within 3 business days of submission Modification Section 2 A 1 "…modified amount must be documented (as an addendum to this chapter) and approved by the Director of the Crime Laboratory Service" Modification Section 3.6 A "At least one complete package unit should be retained (e.g. one brick, bale, bundle, box, etc.). "
01	07/01/2003	Modification Section 4 A "After analysis proceed to destroy the excess quantity in accordance with the provisions of Texas Health and Safety Code §481.160 with or without written authorization from the officer or prosecuting attorney's office and the policy defined in the Laboratory Operation Guide for the destruction of evidence." Modification Section 4 E. "If the evidence is not scheduled for destruction after the analysis, the lab supervisor should make every effort to destroy excess quantity evidence after 45 days of issuance of the report."
02	12/01/2003	Addition to Section 1 "…and Texas DPS Administrative Code Title 37 Part 1."
03	01/01/2005	Minor Revision to Section 2 A #1 Moved previous Section 3.2 to current 3.5 Modification to Section 3.5 A "…the total amount of property or plant in the case and a sufficient quantity, which is at least that amount defined as excess quantity, preserved to provide for discovery." Addition Section 3.5 B providing for criteria of liquid sample.
04	10/10/2006	Minor Revision Modification to Section 1 "Provisions for examination and destruction of ~~excess quantity~~ evidence submitted by a DPS officer and/or a DPS

04	10/10/2006	Task Force that has been identified as excess quantity…." Modification to Section 2 A "Excess quantity ~~evidence~~ is defined as greater than:" Deletion Section 3.1 A regarding before analysis weight. Deletion Section 3.1 B and C regarding examinations of all bundles and determining sufficient number of samples. Addition Section 3.1 A "All evidence packages must be either marked with case number upon receipt or segregated and sampled within five business days of receipt." Addition Section 3.1 B "Conduct examinations as required under Examination of Controlled Substances, Dangerous Drugs and Related Compounds, CS-03-01." Modification to Section 4 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." Deletion Section 4 E "~~If the evidence is not scheduled for destruction after the analysis, the lab supervisor should make every effort to destroy excess quantity evidence after 45 days of issuance of the report.~~"
05	02/09/2009	Major revision – Sections 2, 3.2, and 4 Advisory Board Meeting 11/20/2008

DEPARTMENT OF PUBLIC SAFETY
INTEROFFICE MEMORANDUM

TO: Thomas Ruocco, Chief, Criminal Law Enforcement

DATE: February 13, 2009

FROM: DPJ D. Pat Johnson, Director, Crime Laboratory Service

SUBJECT: Destruction of Excess Quantities of Controlled Substances

It is department policy for officers, upon seizing controlled substances, to submit them to a DPS crime laboratory for analysis, storage, and ultimate destruction. Section 481.160 of the Texas Health and Safety Code (HSC) provides for destruction of excess quantities of controlled substance property and plants as defined by HSC Section 481.151 without a court order before a case's disposition once certain conditions have been met (retain five representative samples and weigh and photograph the entire amount). "Excess quantity" is defined by Title 37, Texas Administrative Code (TAC), Section 13.151 as "more than (A) two kilograms of bulk dry evidence, such as powder; (B) 500 milliliters of bulk liquid evidence, such as a chemical precursor or liquid controlled substance; (C) 200 dosage or abuse units of an item, such as tablets, capsules, liquids, or other items so measured; (D) 50 pounds of bulk packaged marihuana; (E) five individual controlled substance plants, such as marihuana or peyote; or (F) five miscellaneous items of drug or inhalant paraphernalia" unless otherwise modified by a written standard operation procedure (SOP) under 37 TAC 13.157.

Further, 37 TAC 13.157(e)(4) requires that written authorization be obtained from either the prosecutor, submitting peace officer, or submitting law enforcement agency before destroying all or part of each exhibit. This written authorization can be a blanket authority to destroy all or part of each exhibit that meets certain criteria. The Laboratory Manager for each of the department's twelve regional crime laboratories assumes responsibility for the proper destruction of the evidence and follows the laboratory's written destruction SOP as well as applicable statutes, department policy, and rules including the security control measures described in 37 TAC 13.163.

While the DPS crime laboratories receive and examine controlled substance evidence from both non-DPS and DPS officers, it is policy to destroy only DPS evidence and return non-DPS evidence to the submitting agency. When the DPS officer advises the laboratory that a case is being filed in federal court, other arrangements regarding destruction of excess quantities can be made.

REQUEST CLE CHIEF TO OBTAIN DPS DIRECTOR'S APPROVAL
The Crime Laboratory Service requests authorization from DPS management to destroy excess quantities of controlled substance property and plants received from DPS officers which will involve prosecution in state court.

Attachments

Ck. Clark: Approval to destruction of excess quantities of Controlled Substances requires the authorization of DPS management. This policy change was reviewed by CLE legal who advised that this request is within the rules and regulations set forth by the laws listed. I approve this policy revision 2/20/09

Approved - Stanley E. Clark, Director 2-23-09

Preparer

For Ruben A. Rendon, Jr. by Forrest W. Davis Date: 03/03/2009
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 03/03/2009
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	08/18/2000	Original Issue
01	03/03/2009	Minor revision – updated authorization

MICROSCOPIC EXAMINATION OF MARIHUANA

1 Scope

To establish an analytical procedure for the microscopic examination of marihuana, hashish and other plant material.

2 Related Documents

Controlled Substances Worksheet (LAB-CS-01)

Marihuana Worksheet (LAB-CS-02), optional

Examination of Controlled Substances, Dangerous Drugs and Related Compounds (CS-03-01)

3 Safety

1. The plant material, and the 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.
2. Material Safety Data sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.
3. Use proper lifting techniques and caution when handling heavy items.
4. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

4 Equipment, Materials and Reagents

1. Stereoscope
2. Microscope
3. Microscope slides and cover-slips
4. Petroleum Ether or other suitable solvent

5 Practice

5.1 Plant material

1. For leaves, microscopically examine and document the presence of cystolithic hairs (*"bear-claw"* hairs) and glandular hairs on one side of the leaf, conical trichomes or filamentous hairs on the other side of the leaf.
2. Other plant characteristics may be noted. If no leafy material was present, then document the characteristics observed.

    a) Seeds are identified by size, shape and surface pattern. Additionally, the viability of seeds may be determined by germinating a sample of the seeds (CS-03-08).

b) Stalks, the main axis of a plant, are fluted in appearance and covered in hairs. Mature stalks are considered those greater than one-quarter-inch in diameter.

c) Stems, a support structure for another part of the plant such as a leaf or flower, are fluted in appearance and may have hairs on the surface.

5.2 Hashish, oil extracts, ashes, charred material, or residue

1. Microscopically examine the substance for the presence of plant material.
2. If the characteristics of marihuana are not found:

a) Suspend the sample in a suitable solvent.

b) Place the suspension on a microscope slide and cover with a cover-slip.

c) Examine under appropriate magnification.

3. Document the observation(s).

6 Interpretation

A. Microscopic confirmation of the physical characteristics of marihuana constitutes a required examination for the identification of marihuana.

B. A (+) on the microscopic section of the Controlled Substances Worksheet (LAB-CS-01) indicates:

1. For leaves, the following characteristics must be observed:

a) cystolithic hairs, and

b) conical trichomes and/or glandular hairs.

2. For non-leafy material, the following was observed:

a) seeds and/or

b) stems

C. An inconclusive or negative determination may be made at the discretion of the examiner based on the overall appearance of the material.

7 Literature and Supporting Documentation

J. L. Thornton, and G. R. Nakamura. 1972. The Identification of Marihuana. J. Forensic Science Society, 12:461-529.

Preparer

Larry Todsen Date: 06/12/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 06/12/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Major Revision, Text from chapter 4 Deletion 4.2 Sections 1.2-1.4 Modification Section 4.2 with simplification of germination procedure
01	07/01/2003	Modification Section 4.2 Header "Germination of Marihuana Seeds"
02	10/10/2006	Title Change Major Revision, pertaining to • Move Germination procedure to new document. • Optional Marihuana worksheet (LAB-CS-02). • Examine the material and document the presence of cystolithic hairs, conical trichomes, and/or glandular hairs. • Require at least two of three characteristic features to constitute a positive result for microscopic examination of marihuana. • Manner of recording positive results.

PREPARATION AND ANALYSIS OF PEYOTE

1 Scope

To establish a procedure for analysis of peyote.

2 Safety

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.

Material Safety Data sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.

Use proper lifting techniques and caution when handling heavy items.

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

3 Equipment, Materials and Reagents

1. Sonicator
2. Vortex mixer
3. Reagents
   - 0.1 N HCl
   - Petroleum ether
   - NaOH
   - $CHCl_3$
   - 1 M Na_2CO_3
   - 0.2 N H_2SO_4

4 Practice

4.1 Visual Identification

1. Examine the plant material for the following physical characteristics:

   Small, gray-green spineless cactus, approximately 1-3 inches in diameter. 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 H_2SO_4. 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 beaker.
2. Cover with 1 M Na_2CO_3.
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 $CHCl_3$ three times and discard $CHCl_3$.
7. Make extract basic add $CHCl_3$ and filter.
8. The extract may be analyzed by either GC/MS or FTIR.

5 Interpretation

Visual identification is required in order to conclude that Peyote is present.

Proceed with desired analytical testing.

6 Literature and Supporting Documentation

John L. Barbara, “Extraction of Mescaline from Peyote and Subsequent Instrumental Analysis,” Microgram, Volume VIII, No. 12, December, 1975, 182-186.

Preparer

Hector Cadena Date: 05/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 05/01/2003
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Major Revision, Text from chapter 4 Addition Section 4.2 C, Analysis Procedure Addition to Section 5 "Visual identification is required in order to conclude that Peyote is present."
01	07/01/2003	Minor Revision with reference to UV changed to UV/VIS and references to IR changed to FTIR

PREPARATION AND ANALYSIS OF MUSHROOMS

1 Scope

To establish a procedure for preparation and analysis of mushrooms.

2 Safety

1. The plant material and the 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.
2. Material Safety Data sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.
3. Use proper lifting techniques and caution when handling heavy items.
4. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

3 Equipment, Materials and Reagents

1. Sonicator
2. Centrifuge
3. Vortex mixer
4. Laboratory oven
5. Glass wool
6. Reagents
   - Methanol
   - 0.1 *N* HCl
   - Diethyl ether
   - NaOH
   - $CHCl_3$
   - Glacial acetic acid
   - 0.2 *N* H_2SO_4
   - 0.1 *M* H_2SO_4
   - Aqueous base solution
   - BSTFA [*N,O*-bis(trimethylsilyl)trifluoroacetamide]
   - Acetone

4 Practice

4.1 Extraction for TLC or UV/VIS; Preliminary Preparation for GC/MS or Derivatization

1. Place a portion of the mushrooms in methanol. Soak, vortex, or sonicate the extract.

2. Filter out the solids and concentrate the methanol extract. The methanol extract may be analyzed by UV/VIS or TLC.

3. Proceed to GC/MS and/or Derivatization techniques.

4.2 Preparation Techniques for GC/MS

A. Procedure A

1. Add 3 ml diethyl ether to the methanol extract, which will cause a precipitate to form. Agitate the sample.

2. Centrifuge the extract and discard the supernatant liquid. Wash the yellow precipitate with 2 ml diethyl ether; centrifuge and discard the supernatant liquid.

3. Add 2-3 ml methanol and continue with desired tests.

B. Procedure B

1. Evaporate the methanol extract to dryness. Add approximately 1 ml H_2O, two drops of concentrated HCl and 2 ml chloroform.

2. Vortex mixture for approximately 2 minutes and centrifuge.

3. Transfer aqueous layer to a clean test tube and add concentrated NH_4OH drop-wise until the solution is basic to pH paper, then add 0.5 ml chloroform.

4. Vortex mixture for approximately 2 minutes and centrifuge.

5. Dry the chloroform solution by passing it through anhydrous sodium sulfate.

6. Concentrate to a small volume for analysis by GC/MS.

C. Procedure C

1. Place a portion of the mushrooms in 0.2 *N* H_2SO_4. Soak at least 30 minutes.

2. Filter out the solids, make the acid extract basic to pH paper, and extract with chloroform. The extract may be analyzed by GC/MS.

D. Procedure

1. Powder dried mushrooms. Add 15-20 ml H_2O.

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

3. Allow to soak at least 10 minutes.

4. Filter solution through glass wool to remove plant material.

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

6. Extract solution with $CHCl_3$. The extract may be analyzed by GC/MS.

7. Back extract with 0.1*M* H_2SO_4. The extract may be analyzed by UV/VIS.

4.3 Derivatization Techniques

A. Procedure A

1. Soak mushroom sample in methanol overnight. Centrifuge the mixture and collect the supernatant liquid.
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 TLC.
5. Place a portion of the liquid concentrate into a vial or insert.
6. Evaporate the liquid extract to dryness with a stream of dry air.
7. Add 100 µl BSTFA to the dried residue.
8. Cap vial and incubate approximately 30 min at 90-100^{o} C.
9. Analyze by GC/MS. After the injection of the sample, clean the syringe with ethyl acetate.

B. Procedure B

1. Evaporate a methanol extract to dryness.
2. Add 1-2 drops BSTFA.
3. Incubate 5 to 10 minutes at 90-100^{o} C.
4. The extract may be analyzed by GC/MS.
5. After the injection of the sample, clean the syringe with ethyl acetate.

5 Interpretation

Proceed with desired analytical testing.

6 Literature and Supporting Documentation

Robert Earl Lee, “A Technique for the Rapid Isolation and Identification of Psilocin from Psilocin/Psilocybin-containing Mushrooms,” *Journal of Forensic Science*, July, 1985, 931-941.

Preparer

Hector Cadena Date: 05/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 05/01/2003
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Major Revision, Text from chapter 4 Addition Section 4.2 D, Analysis Procedure Addition Section 4.3, Derivatization Techniques
01	07/01/2003	Minor Revision with title and references to UV changed to UV/VIS

DERIVATIZATION OF LSD

1 Scope

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

2 Safety

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

3 Equipment, Materials and Reagents

1. Sonicator
2. Centrifuge
3. Vortex mixer
4. Laboratory oven
5. Autosampler vials (or similar) with screw caps
6. Reagents
   - 0.2 *N* H_2SO_4
   - Conc. aqueous NaOH solution
   - BSTFA [*N,O*-bis(trimethylsilyl)trifluoroacetamide]
   - Chloroform
   - Ethyl acetate

4 Practice

1. Place a number of abuse units in dilute aqueous acid and sonicate for at least 15 minutes. The acid extract may be analyzed by UV/VIS.
2. Make the extract basic (using conc. NaOH) and extract with chloroform ($CHCl_3$). The extract may be analyzed by TLC.
3. Evaporate the $CHCl_3$ 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^{o}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 and Supporting Documentation

Shumate, Karen, "Preparation and Identification of the Trimethylsilylderivative of LSD by GC-MS, For DPS in-house publication, Nanogram, March, 1992.

Harper, Charles W., “Silylation and Acylation Derivatives for GLC and GLC-MS Drug Analysis”, Microgram, Volume XII, No. 4, p 82-86.

Preparer

Hector Cadena Date: 05/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 05/01/2003
Quality Assurance

Version #	Issue Date	Brief Description of Change(s)
00	12/01/2002	Original Issue
01	07/01/2003	Minor Revision, references to UV changed to UV/VIS and derivitization corrected to derivatization Addition Section 3 6. reagent "Ethyl Acetate" Addition Section 6 Literature Citation

DERIVATIZATION OF GHB

1 Scope

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

2 Safety

Material Safety Data sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.

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

3 Equipment, Materials and Reagents

1. Gas Chromatograph Mass Spectrometer
2. Autosampler vials (or similar) with screw caps
3. Hot plate or oven (temperature 80 – 90^{o}C)
4. 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.
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.

   Note: You may substitute BSTFA with TMCS for steps 3 and 4.
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

Proceed to GC/MS analysis.

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

6 Literature

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

Stephens, Debra L., The Rave Party Drug: GHB and its Analysis

Johnson, Ruth H. and Bussey, Janet L., Assay Procedure for the Sodium Salt of Gamma Hydroxybutyric Acid," FDA **Laboratory Information Bulletin** No. 3532.

Preparer

Hector Cadena Date: 11/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 11/01/2003
Quality Assurance

Version #	Issue Date	Brief Description of Change(s)
00	07/01/2003	Original Issue
01	12/01/2003	Modification to Section 4 #6 “Remove from oven and let test tube cool.“

GERMINATION OF MARIHUANA SEEDS

1 Scope

To establish a procedure for germination of marihuana seeds.

2 Related Documents

Controlled Substances Worksheet (LAB-CS-01)

Marihuana Worksheet (LAB-CS-02), optional

Examination of Controlled Substances, Dangerous Drugs and Related Compounds, CS-03-01

Microscopic Examination of Marihuana (CS-03-03)

3 Safety

1. The plant material, dust, and mold may trigger allergic reactions, which may require susceptible personnel to take precautionary measures, such as wearing masks, respirators and gloves.
2. Material Safety Data sheets are available in the laboratory if additional information is needed about any of the chemicals used in the analytical procedure.
3. Use proper lifting techniques and caution when handling heavy items.
4. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

4 Equipment, Materials and Reagents

1. Stereoscope
2. Filter paper or equivalent
3. Plastic box with lid or other appropriate container

5 Practice

1. Document if seeds are identified by color and appearance as being marihuana.
2. Select at least 100 seeds at random.
3. Place seeds between moistened filter paper or equivalent, and place in an appropriate container.
4. Incubate at room temperature for up to 10 days.
5. Document if any seeds germinate.

6 Interpretation

A. Marihuana seeds typically are green to brown, ovoid, and have a tortoise shell (reticulated) pattern.

B. If any seeds germinate, it is determined that the seeds are capable of beginning germination.

7 Literature and Supporting Documentation

Marihuana - Its Identification, U.S. Treasury Department, Bureau of Narcotics, U.S. Government Printing Office, Washington D.C. 1948.

Preparer

Larry Todsen Date: 06/12/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 06/12/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	10/10/2006	Original Issue, text from CS-03-03 Modification Section 5 #2 regarding to sampling for germination procedure

CHEMICAL SCREENING SPOT TESTS – OVERVIEW

1 Scope

To describe the chemical screening procedures, commonly referred to as chemical spot tests, for preliminary tests of controlled and non-controlled substances.

2 Safety

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.

Refer to MSDS for additional safety information for specific chemicals.

3 Equipment, Materials and Reagents

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

4 Standards, Controls and Calibration

Each reagent must be labeled with the name of the solution or reagent. The analyst's initials and the date prepared must be recorded on the label or in an appropriate logbook.

Freshly prepared reagents will be quality tested with known reference standards and the results recorded in a retrievable logbook.

Unless otherwise specified, performance of reagents will be verified monthly and the results of the checks placed in a logbook. 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.

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

All spot tests are presumptive in nature and serve only as a guide for an analyst's analytical scheme.

Adulterants and complex mixtures may produce reactions that interfere with the interpretations.

6 Advantages

Spot tests provide a quick and easy method for determining what a sample might contain..

Spot tests can assist in the determination of appropriate analytical processing, collection of appropriate samples, and grouping samples for uniformity testing.

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

Concurrence

C. *Glen Johnson* Date: 11/01/2002
Quality Assurance Coordinator

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
	12/07/2001	Change all instances of deionized H2O to purified H2O
01	12/01/2002	Major Revision; Moved Text from Chapter 5 Text from each Test Separated into individual documents Addition Section 4 "Freshly prepared reagents will be quality tested with known reference standards and the results recorded in a retrievable logbook. Unless otherwise specified, performance of reagents will be verified monthly and the results of the checks placed in a logbook. 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. 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. "

MARQUIS TEST

1 Reagents/Chemicals

- Conc. Sulfuric acid
- Formaldehyde Solution (approx. 37% Formaldehyde)

Marquis Reagent

Add 1 mL formaldehyde solution to 9 mL conc. sulfuric acid.

Shelf life is limited.

Quality-test reagent with amphetamine, methamphetamine, or an opiate.

2 Procedure

1. Combine a small amount of sample with a few drops of Marquis Reagent.
2. Record any resulting color reaction(s).

3 Interpretation

A. Various colors representing the whole of the visible spectrum may be given by a large number of compounds. Additional results or interpretations may be found in Stevens (1986).

B. A reaction which forms an orange color indicates the possible presence of amphetamine or methamphetamine.

C. A reaction which forms a black color indicates the possible presence of Dextromethorphan, MDA or its analogues.

D. A reaction which forms a dark purple color indicates the possible presence of heroin, opiates, methocarbamol, or guaifenesin.

E. A reaction which forms a red color indicates the possible presence of salicylates.

F. The color which appears must be documented on the examination worksheet.

4 Literature and Supporting Documentation

H. M. Stevens, 1986. "Colour Tests" in *Clarke's Isolation and Identification of Drugs*, ed. A. C. Moffat (London: The Pharmaceutical Press) 128-147.

Preparer

Larry Todsen Date: 04/24/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 04/24/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Modification Section 1 "…reagent will be quality tested every two weeks." "Quality-test reagent with amphetamine, methamphetamine, or an opiate."
01	07/01/2003	Minor Revision, with reference to 37% Formaldehyde and Formaldehyde solution Addition Section 3 "Dextromethorphan"
02	01/01/2005	Modification to Section 1 "….Shelf life is limited. ~~This reagent will be quality tested every two weeks~~…."
03	10/10/2006	Modification to Section 1 "Add 1 mL ~~drop of~~ formaldehyde solution to ~~1~~ 9 mL conc. sulfuric acid." Deletion from Section 4 literature citation for Jones et al.

COBALT THIOCYANATE (COCAINE) TESTS

1 Reagents/Chemicals

- Cobalt thiocyanate
- Glycerin
- Purified H_2O
- Concentrated HCl or other acid
- Chloroform ($CHCl_3$)

Acceptable Alternative Test CoSCN Reagents:

Scott Reagent

Dissolve 2 g cobalt thiocyanate in 100 mL H_2O and dilute with 100 mL glycerin.

2% Cobalt Thiocyanate with Glycerin Reagent

Dissolve 2 g cobalt thiocyanate in 50 mL H_2O and dilute with 50 mL glycerin.

2% Cobalt Thiocyanate Reagent

Dissolve 2 g cobalt thiocyanate in 100 mL H_2O.

Quality-test reagent with cocaine standard.

2 Procedure

1. Combine a small amount of sample with the reagent. If a positive result is obtained, the analyst may stop and record any observations.
2. Add acid drop-wise to the sample until color disappears. The analyst may stop and record any observations.
3. Approximately five drops of $CHCl_3$ may be added to extract any soluble complexes. Record any observations.

3 Interpretation

An appropriate notation on the worksheet documents that the addition of the reagent resulted in a blue color (+), the addition of the acid resulted in a pink color, and the addition of the $CHCl_3$ resulted in a blue color (+) in the lower layer. This indicates a cocaine salt may be present.

An appropriate notation on the worksheet documents the addition of the reagent resulting in no color change (-) until a drop of acid has been added to the solution, which then resulted in a blue color (+). This indicates that cocaine base may be present.

4 Literature and Supporting Documentation

Scott LJ. Specific Field Test for Cocaine. 1973. Microgram. 6, pp. 179-181.

Preparer

Ruben A. Rendon, Jr. Date: 11/21/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 11/21/2008
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Rename test to Cocaine Modification section 1 added two alternative test reagents Addition section 2, 1 and 2 "Record any observation. If positive result obtained the analyst may stop." Addition section 2, 4 "Record any observations."
01	07/01/2003	Minor revision, Section 2 Modification Section 2 3. "…may be added…."
02	01/01/2005	Rename Document
03	05/01/2005	Rename Document
04	02/09/2009	Major revision – Section 2 Advisory Board 11/20/2008

SODIUM NITROPRUSSIDE TEST

1 Reagents/Chemicals

- Sodium Nitroprusside
- Purified H_2O
- Acetaldehyde
- 1*M* Sodium Hydroxide (NaOH)
- 1*M* Sodium Carbonate (Na_2CO_3)

SNP Reagent

Dissolve 0.09 g sodium nitroprusside in a mixture of 1 mL acetaldehyde and 9 mL H_2O.

Quality-test reagent with a methamphetamine standard.

2 Procedure

1. Combine a small amount of sample with a few drops of SNP Reagent.
2. Add a few drops of 1*M* Na_2CO_3 (or NaOH) to the sample.
3. Record any observations.

3 Interpretation

A positive indication on the worksheet means a reaction that forms a blue color (+) which indicates the possible presence of secondary amines, such as methamphetamine.

4 Literature and Supporting Documentation

Feeney, F. S. Alcohol and Tobacco Lab, IRS, Treasury Dept. Washington, DC.

Feigl, Fritz. 1956. Spot tests in Organic Analysis, 5th Edition, Elsevier Publishing Co.

Preparer

Raymond A. Waller, Jr. Date: 10/04/2004
Controlled Substance Advisory Board Chair

Concurrence

Forrest Davis Date: 10/04/2004
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 2, 3 "Record any observations."
01	12/01/2003	Modification to Section 4 Literature added and deleted
02	01/01/2005	Minor Revision Modification to Section 1 reagent "Dissolve ~~0.1~~0.09 g sodium nitroprusside…."

SODIUM m-PERIODATE (SMP) TEST

1 Reagents/Chemicals

- Sodium Metaperiodate
- 1 *M* NaOH
- 1 *M* Sodium Carbonate

Quality-test with ephedrine or pseudoephedrine.

2 Procedure

1. Combine a small amount of sample and dry $NaIO_4$.
2. Add a few drops of 1 *M* Na_2CO_3 or NaOH.
3. Record any observations.

3 Interpretation

A positive indication on the worksheet means the reaction resulted in a cherry odor (+), which is due to the formation of benzaldehyde and indicates the possible presence of ephedrine, pseudoephedrine, or phenylpropanolamine.

A brown color indicates the possible presence of acetaminophen.

4 Literature and Supporting Documentation

Ginn, W. L. 1988. DPS Training Manual.

Preparer

Hector Cadena Date: 11/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 11/01/2003
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 3 "…cherry odor (+), which indicates the possible presence of ephedrine, pseudoephedrine, or phenylpropanolamine."
01	07/01/2003	Modification Section 2 "…which is due to the formation of benzaldehyde and indicates the possible presence of…."
02	12/01/2003	Modification to Section 4 Literature added and deleted

FERRIC CHLORIDE TEST

1 Reagents/Chemicals

- Ferric Chloride, $FeCl_3 \cdot 6H_2O$
- Purified H_2O

5% Ferric Chloride Reagent: Dissolve 0.83 g $FeCl_3 \cdot 6H_2O$ in 10 mL H_2O.

Quality-test reagent with GHB or aspirin.

2 Procedure

1. Combine a small amount of sample and a few drops of 5% Ferric Chloride reagent.
2. Record any observations.

3 Interpretation

A reaction that forms an orange-brown color indicates the possible presence of GHB.

A reaction that forms a dark purple color indicates the possible presence of salicylates.

A reaction that forms a bluish-gray color indicates the possible presence of acetaminophen.

The resulting color must be indicated on the examination worksheet.

4 Literature and Supporting Documentation

H. M. Stevens, “Colour Tests” in *Clarke's Isolation and Identification of Drugs*, ed. A. C. Moffat (London: The Pharmaceutical Press) 128-147.

Frommhold, S., and C. Busby. 1996. GHB: A brief review with a few case histories. SWAFS Journal, 18(2):5-15.

Preparer

Hector Cadena Date: 11/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 11/01/2003
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 1 "$FeCl_3 \cdot 6H_2O$"
01	07/01/2003	Minor Revision with reference to GHB (gamma hydroxybutyrate) Modification Section 1 Reagent preparation "Dissolve 0.83 g $FeCl_3 \cdot 6H_2O$ in 10 ml H_2O
02	12/01/2003	Modification to Section 4 Literature Added and Deleted

WINTERGREEN TEST

1 Reagents/Chemicals

- Sodium Hydroxide
- Potassium Hydroxide
- Methanol

10% Sodium Hydroxide in methanol: Dissolve 1 g sodium hydroxide in 10 mL methanol.

10% Potassium Hydroxide in methanol: Dissolve 1 g potassium hydroxide in 10 mL methanol.

Quality-test reagents with a cocaine standard.

2 Procedure

1. Combine a small amount of sample to a few drops of either 10% NaOH in methanol or 10% KOH in methanol reagent.

2. Record any observations.

3 Interpretations

A positive indication on the worksheet means the reaction resulted in a wintergreen odor (+), which is due to production of methyl benzoate, and which indicates the possible presence of cocaine.

4 Literature and Supporting Documentation

Grant, F. W., W. C. Martin, and R. W. Quackenbush. 1975. A simple field test for cocaine not relying on cobalt thiocyanate. Microgram 8:10-11.

Preparer

Hector Cadena Date: 11/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 11/01/2003
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 2, 2 "Record any observations."
01	12/01/2003	Modification to Section 4 Literature Added and Deleted

COBALT NITRATE TEST

1 Reagents/Chemicals

- Cobalt nitrate
- Isopropylamine
- 95% ethanol

1% Cobalt nitrate in ethanol reagent: Add 1 g cobalt nitrate to 100 mL ethanol.

5% Isopropylamine in ethanol reagent: Add 5 g isopropylamine to 100 mL ethanol.

Quality-test reagent with a gamma-hydroxybutyrate or barbiturate standard.

2 Procedure

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

3 Interpretation

A purple color upon addition of 1% cobalt nitrate in ethanol indicates the possible presence of gamma-hydroxybutyrate (GHB+).

A purple color which only forms after also adding 5% isopropylamine in ethanol indicates the possible presence of barbiturates (Barb+).

4 Literature and Supporting Documentation

W. J. Stall, “The Cobalt Nitrate Color Test,” *Microgram* 13 (1980): 40-43.

H. M. Stevens, 1986. “Colour Tests” in *Clarke's Isolation and Identification of Drugs*, ed. A. C. Moffat (London: The Pharmaceutical Press) 128-147.

Johns, S. H. et. al. “Spot Tests: A Color Chart Reference for Forensic Chemists,” *Journal of Forensic Sciences* 24 (1979) 631-649.

Frommhold, S., and C. Busby. 1996. GHB: A brief review with a few case histories. SWAFS Journal, 18(2):5-15.

Preparer

Hector Cadena Date: 11/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 11/01/2003
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 2, 2 and 4 "Record any observations."
01	12/01/2003	Addition to Section 4 additional references

p-DMABA TEST

1 Reagents/Chemicals

- 95% Ethanol
- p-Dimethylaminobenzaldehyde
- Conc. HCl

p-DMABA reagent: Dissolve 0.1 g p-dimethylaminobenzaldehyde in 9.5 ml ethanol. Add 0.5 ml conc. HCl.

Quality-test reagent with benzocaine, procaine, or LSD.

2 Procedure

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

3 Interpretation

A reaction which forms a bright yellow color indicates the possible presence of procaine or benzocaine.

A reaction which forms a purple color indicates the possible presence of LSD.

The resulting color must be indicated on the examination worksheet.

4 Literature and Supporting Documentation

H. M. Stevens, “Colour Tests” in *Clarke's Isolation and Identification of Drugs*, ed. A. C. Moffat (London: The Pharmaceutical Press) 128-147.

Johns, S. H. et. al. “Spot Tests: A Color Chart Reference for Forensic Chemists,” *Journal of Forensic Sciences,* 24 (1979): 631-649.

Basic Training Program for Forensic Drug Chemists, U. S. Dept. of Justice., 2nd Edition.

Preparer

Hector Cadena Date: 05/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 05/01/2003
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 2, 2 "Record any observations."
01	07/01/2003	Minor Revision with reference to Conc. HCl

WEBER TEST

1 Reagents/Chemicals

- Fast Blue B
- Conc. HCl
- Purified H_2O

0.1% Fast Blue B: Dissolve 0.1 g Fast Blue B in 100 mL H_2O.

Prepare this reagent fresh and quality-test with psilocin or a sample of mushroom shown to contain psilocin before use.

2 Procedure

1. Combine a small amount of sample or methanol extract of the sample and a few drops of 0.1% Fast Blue B wait approximately one minute.

2. Add one volume of conc. HCl.

3. Record any observations.

3 Interpretations

A positive reaction for psilocin is indicated if a red color forms after adding the Fast Blue B reagent, and if after adding HCl the color changes to blue (appropriate color sequence = Weber+).

4 Literature and Supporting Documentation

A. S. Garrett, Clemens, J. Gaskill, “The Weber test: a color test for the presence of psilocin in mushrooms,” *SWAFS Journal,* 15 (1993): 44-45.

Preparer

Larry Todsen Date: 04/24/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 04/24/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Modification Section1 "Prepare this reagent fresh and quality-test with psilocin before use." Addition section 2, 3 "Record any observations."
01	07/01/2003	Minor Revision with reference to conc. HCl
02	10/10/2006	Modification Section 1 "…with psilocin or a sample of mushroom shown to contain psilocin before use."

DUQUENOIS-LEVINE TEST

1 Reagents/Chemicals

- Vanillin
- 95% Ethanol
- Acetaldehyde
- Conc. HCl
- Chloroform
- Petroleum ether

Duquenois Reagent: Add 0.4 g vanillin and 5 drops acetaldehyde to 20 mL 95% ethanol.

Quality-test the reagent with a known sample of marihuana or tetrahydrocannabinol.

2 Procedure

1. Place a small amount of plant material in a testing container. Either proceed directly to the next step or extract the plant material with petroleum ether. If extracted, discard the plant material, and evaporate to dryness.
2. Add one volume of the Duquenois reagent and wait approximately one minute. (It is not necessary to wait as long with the extract.)
3. Add one volume of conc. HCl.
4. Add one volume of $CHCl_3$.
5. Record any observations.

3 Interpretation

A. A blue to violet color after the addition of HCl to the mixture of Duquenois reagent and plant material or extract is a positive reaction and indicates the possible presence of THC.

B. After adding $CHCl_3$ and mixing, a purple color in the organic (lower) layer is a positive reaction for the possible presence of THC.

C. A positive result indicates that the components (cannabinoids, including THC) unique to marihuana, marihuana residue, or hashish are present.

D. A positive (+) indication on the worksheet means the test resulted in a blue to violet color after the addition of the HCl and Duquenois reagents and that the $CHCl_3$ layer yielded a purple color.

4 Literature and Supporting Documentation

C. G. Pitt, et. al. “The Specificity of the Duquenois Color Test for Marijuana and Hashish,” *Journal of Forensic Science,* 17 (1972): 693-700.

Preparer

Larry Todsen Date: 04/24/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 04/24/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 2, 5 "Record any observations."
01	07/01/2003	Minor Revision with reference to conc. HCl Modification Section 3 "After adding $CHCl_3$ and mixing, a purple color in the organic (lower) layer is a positive reaction for the possible presence of THC."
02	10/10/2006	Modification Section 1 "…of marihuana or tetrahydrocannabinol."

SULFURIC ACID TEST

1 Reagents/Chemicals

- Conc. Sulfuric acid

Quality-test reagent with a known sample of steroid.

2 Procedure

1. Combine a small amount of sample and a few drops of conc. Sulfuric acid.
2. Record any observations.
3. A UV light may be used to aid visualization of a color change.

3 Interpretation

An orange or yellow color may indicate the possible presence of a steroid.

Note: Certain oils may give an orange result as mentioned in the reference.

The resulting color must be indicated on the examination worksheet.

4 Literature and Supporting Documentation

H. M. Stevens, 1986: “Colour Tests” in *Clarke's Isolation and Identification of Drugs*, ed. A. C. Moffat (London: The Pharmaceutical Press) 128-147.

Preparer

Ruben A. Rendon, Jr. Date: 11/21/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 11/21/2008
Quality Assurance

Version #	Issue Date	Brief Description of Change(s)
00	12/01/2002	Original Issue
01	07/01/2003	Minor revision with reference to conc. Sulfuric acid
02	02/09/2009	Major revision – Section 3 Advisory Board 11/20/2008

FORMALDEHYDE-SULFURIC ACID TEST

1 Reagents/Chemicals

- Conc. Sulfuric Acid
- Formaldehyde Solution (i.e. 37% Formaldehyde)

Formaldehyde-Sulfuric Reagent: Add 6 volumes of formaldehyde solution to 4 volumes conc. Sulfuric acid. Keep the pipette tip just below the surface during the addition; stir and (if necessary) cool the mixture.

Quality-test reagent with a known sample of benzodiazepines.

2 Procedure

1. Combine a small amount of sample and a few drops of formaldehyde-sulfuric test reagent in a test tube.
2. Heat to approximately 100^{o} C for approximately a minute.
3. Record any observations.

3 Interpretation

Benzodiazepines generally give an orange color.

The resulting color must be indicated on the examination worksheet.

4 Literature and Supporting Documentation

H. M. Stevens, 1986: “Colour Tests” in *Clarke's Isolation and Identification of Drugs*, ed. A. C. Moffat (London: The Pharmaceutical Press), 128-147.

Preparer

Hector Cadena Date: 05/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 05/01/2003
Quality Assurance

Version #	Issue Date	Brief Description of Change(s)
00	12/01/2002	Original Issue
01	07/01/2003	Minor revision with reference to conc. Sulfuric acid and formaldehyde solution

LIEBERMANN TEST

1 Reagents/Chemicals

- Sodium nitrite
- Sulfuric Acid

Liebermann Reagent

Carefully add 5 g sodium nitrite to 50 mL sulfuric acid with cooling and swirling. Perform the addition in the hood, as toxic nitrogen oxides are produced.

Quality-test the reagent with a known sample of codeine, methylphenidate, ephedrine, mescaline, or d-propoxyphene.

2 Procedure

1. Combine a small amount of sample and a few drops of Liebermann Reagent.
2. Heat to approximately 100°C for approximately one minute.
3. Record any observations.

3 Interpretation

Various colors may be produced by a large number of different compounds. Additional results or interpretations may be found in Stevens (1986).

The resulting color must be indicated on the examination worksheet.

4 Literature and Supporting Documentation

H. M. Stevens, 1986: "Colour Tests" in *Clarke's Isolation and Identification of Drugs*, ed. A. C. Moffat (London: The Pharmaceutical Press), 128-147.

Preparer

Raymond A. Waller, Jr. Date: 10/04/2004
Controlled Substance Advisory Board Chair

Concurrence

Forrest Davis Date: 10/04/2004
Quality Assurance

Version #	Issue Date	Brief Description of Change(s)
00	12/01/2002	Original Issue
01	01/01/2005	Modification to Section 1 adding Codeine as option for Quality Test

PHOSPHORUS TEST

1 Scope

A modified Sigma Diagnostics procedure for colorimetric detection of phosphorus is provided.

2 Reagents/Chemicals

- Phosphorus Detection Kit, Sigma Diagnostics #670-A, which contains Acid Molybdate Solution, Fiske & Subbarow Reducer Solution, and Phosphorus Standard Solution
- 0.2 *N* Sulfuric acid
- Purified H_2O

Quality-test reagent with a known sample of phosphorus.

3 Procedure

1. Combine approximately 0.20 g sample, 1 ml 0.2 *N* Sulfuric acid, and 1 ml purified H_2O. Mix the sample with the liquid and let stand for 2-3 minutes.
2. Centrifuge the solution until a relatively clear liquid is present.
3. In a separate test tube, combine 1 ml of the supernatant, 1.5 ml purified H_2O, and 0.5 ml Acid Molybdate Solution. Mix the contents of the test tube.
4. Add 4 drops Fiske & Subbarow Reducer Solution.
5. Mix the contents by inversion and let stand.
6. Record any observations.

4 Interpretation

A positive result for the presence of phosphorus is the formation of a pale blue color followed by a more intense royal blue color (+). The reaction may occur over a period of about ten minutes.

5 Literature and Supporting Documentation

Sigma Diagnostics, *Phosphorus, Inorganic,* Procedure No. 670.

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

Concurrence

C. *Glen Johnson* Date: 11/01/2002
Quality Assurance Coordinator

Version #	Issue Date	Brief Description of Change(s)
00	12/01/2002	Original Issue

AMMONIUM TEST PAPER

1 Scope

A modified procedure for detection of ammonium.

2 Safety

These test strips are poisonous and should be handled with gloves.

3 Reagents/Chemicals

- Gallard-Schlesinger Ammonium Test Paper #90722
- 10% Sodium Hydroxide

4 Procedure

4.1 For testing aqueous sample:

1. Use forceps to remove test strip from packaging. The test strips are poisonous.
2. Apply several drops of the aqueous sample.
3. Apply several drops of 10% NaOH solution to test strip.
4. Record any observations.

4.2 For testing anhydrous sample from tank:

1. Use forceps to remove test strip from packaging. The test strips are poisonous.
2. Drop purified H_2O onto the test strip.
3. Expose test strip to gas (1 to 25 seconds).
4. Apply several drops of 10% NaOH solution to test strip.
5. Record any observations.

5 Interpretation

A positive reaction for the presence of ammonium is indicated by a color change from white to brown-yellow.

6 Literature and Supporting Documentation

Gallard-Schlesinger Industries, Inc. Rapid Tests catalog, 35th edition, 2003.

Preparer

Hector Cadena Date: 11/01/2003
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 11/01/2003
Quality Assurance

Version #	Issue Date	Brief Description of Change(s)
00	12/01/2003	Original Issue

CHLOROPHENOL RED: MODIFIED SCHWEPPE'S TEST

1 Reagents/Chemicals

Chlorophenol Red solution

40 mg Chlorophenol Red in 100 mL of water, adjusting the pH to 7.0 with 0.1 *N* Sodium Hydroxide

Modified Schweppe's Reagent - Solution A

2 g Dextrose in 20 mL water

Modified Schweppe's Reagent - Solution B

2.4 g Aniline Hydrochloride in 20 mL Methanol

1.1 Procedure for Modified Schweppe's Reagent

1. Mix Solution A and Solution B and dilute to 80 mL with Methanol.
2. Store the Modified Schweppe's Reagent in an amber bottle and refrigerate to retard decomposition.

1.2 Procedure for mixed reagent

1. Test the individual solutions against a blank tap-water sample.
2. Mix the Chlorophenol Red solution and the Modified Schweppe's Reagent using a 3:1 ratio (Chlorophenol Red solution:Modified Schweppe's Reagent).
3. The mixed reagent is stable for up to 3 weeks in an amber bottle on the bench.
4. Test the mixed reagent against a blank tap-water sample. If the result of the test with the blank tap-water sample is a color change to brown, the solutions will need to be remade.

2 Procedure

1. Add approximately 0.5 mL of liquid sample or a small amount of powder sample to a test tube.
2. Check the pH. It is necessary for the pH to be carefully adjusted to 5-8.
3. Add 2 drops of the mixed reagent and swirl.
4. Record any observations.

3 Interpretation

A reaction that forms an orange or red color indicates the possible presence of GHB.

4 Literature and Supporting Documentation

Smith, P. R., and J. S. Bozenko, Jr. 2002. New presumptive tests for GHB. Microgram, 35:10-15.

Preparer

Hector Cadena Date: 11/01/2003
Controlled Substance Advisory Board Chair

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Version #	Issue Date	Brief Description of Change(s)
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JANOVSKY TEST

1 Reagents/Chemicals

- m-dinitrobenzene
- Potassium hydroxide
- Absolute ethanol

Janovsky Solution A

- 2% m-dinitrobenzene: Add 2 g m-dinitrobenzene to 100 mL absolute ethanol.

Janovsky Solution B

- 5*N* potassium hydroxide: Add 28.05 g potassium hydroxide to 100 mL purified water.

Quality-test reagents with Ketamine and/or Flunitrazepam.

2 Procedure

1. Combine equal amounts of Janovsky Solution A and B in an appropriate container.
2. Add a small amount of sample.
3. Record any resulting color reaction(s).

3 Interpretation

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.

A reaction which forms an initial strong purple color and fades to brown indicates the possible presence of Flunitrazepam.

The color which appears must be documented on the examination worksheet.

4 Literature and Supporting Documentation

Rucker, C. L. 1998. Chemical screening and identification techniques for Flunitrazepam. Microgram, 31(7):198-205.

Shadan, A., Rahin, R. Presumptive test for Ketamine by the Janovsky Reagent. Buletin Kualiti dan Teknikal Bil B, Keluaran, 2005; (3):5-9.

Preparer

Ruben A. Rendon, Jr. Date: 03/03/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 03/03/2008
Quality Assurance

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00	01/01/2005	Original Issue
01	05/05/2008	Major revision – Section 4 Advisory Board 02/27/2008

CHEMICAL SCREENING MICROCRYSTALLINE TESTS – OVERVIEW

1 Scope

To describe procedures for the presumptive identification of controlled and non-controlled substances using polarized-light microscopy and microcrystalline reagents.

2 Safety

Microcrystalline tests may use a variety of corrosive, caustic, or other dangerous chemicals. Caution should always be practiced, and appropriate personal protective equipment used.

Refer to MSDS for additional safety information for specific chemicals and proper disposal.

3 Equipment, Materials and Reagents

- Polarizing microscope with analyzer
- Glass slides, including depression well slides
- Pipettes and assorted dropper bottles and other containers for the reagents
- Reagents appropriate to the specific microcrystalline tests.

4 Standards, Controls and Calibration

Each reagent must be labeled with the name of the solution or reagent. The analyst's initials and the date prepared must be recorded on the label or in an appropriate logbook.

Unless otherwise specified, performance of reagents will be verified monthly and the results of the checks placed in a logbook. 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.

It is the responsibility of the analyst to determine if these 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

Microcrystalline tests are not suitable as a means for the confirmation of the identification of an unknown substance.

The presence of other compounds, such as impurities or cutting agents, can inhibit the growth of the crystals of the controlled substance and lead to deformities or irregular shapes.

6 Advantages

Simple method for differentiation of optical isomers.

Requires very little sample for a successful test.

Can be used as a quick method of screening substances.

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

Concurrence

C. _Glen Johnson_ Date: 11/01/2002
Quality Assurance Coordinator

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
	12/07/2001	Change all instances of deionized H2O to purified H2O
01	12/01/2002	Minor Revision; Text from Chapter 5 Addition Section 4 "Unless otherwise specified, performance of reagents will be verified monthly and the results of the checks placed in a logbook. 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. " Each Test Separated into individual documents

GOLD BROMIDE MICROCRYSTAL TEST

1 Reagents/Chemicals

- Gold trichloride
- Hydrobromic acid (40%)
- Sulfuric acid (96%)
- Phosphoric Acid (85%)
- Purified H_2O

Gold bromide stock solution: Dissolve 1.0 g gold trichloride in 1.5 ml 40% hydrobromic acid, and add 28.5 ml dilute sulfuric acid (2 parts of conc. sulfuric acid to 3 parts water).

Gold bromide working solution: Mix one part by volume of the stock solution and two parts by volume 85% phosphoric acid.

Storage: Keep tightly capped and store at room temperature.

Quality-test reagent with cocaine.

2 Procedure

1. Place a small drop of Gold Bromide reagent (working solution) on a glass slide.
2. Add a small amount of the unknown sample to the reagent.
3. Record any observations.

3 Interpretation

Compare the shape/structure of crystals produced with crystals from known material and with reference to literature descriptions.

A crystal with four-pointed star with approximately 45^{o} angles indicates possible presence of cocaine (AuBr+).

4 Literature and Supporting Documentation

Jay A. Siegel, “Forensic Identification of Controlled Substances,” in Forensic Science Handbook, Volume II, ed. Richard Saferstein (Englewood Cliffs, New Jersey: Prentice-Hall, 1988), 80-81.

Charles C. Fulton, Modern Microcrystal Tests for Drugs, (John Wiley & Sons, Inc. 1969)

E. C. G. Clarke, “Microcrystal Tests,” in *Isolation and Identification of Drugs*, ed. E. C. G. Clarke (London, William Clowes and Sons, 1969).

Preparer

Hector Cadena Date: 11/01/2002
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Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 3 "A crystal with four-pointed star with approximately 45º angles indicates possible presence of cocaine (AuBr+)."

GOLD CHLORIDE MICROCRYSTAL TEST

1 Reagents/Chemicals

- Gold chloride ($HAuCl_4 \cdot 3H_2O$)
- Phosphoric acid, 85%
- Sodium hydroxide, 10%
- Purified H_2O

Gold Chloride Reagent: Dissolve 1.0 g gold chloride in a mixture of 7 ml 85% phosphoric acid and 14 ml H_2O.

Storage: Keep tightly capped and store at room temperature.

Quality-test reagent with methamphetamine.

2 Procedures

2.1 Direct

1. Place a small drop of gold chloride reagent on the glass slide.
2. Add a small amount of the unknown.
3. Stir the sample/reagent mixture.
4. Record any observations.

2.2 Volatile Hanging Drop

1. Place a small amount of the unknown in a well of a microwell slide.
2. Add a small amount of 10% sodium hydroxide.
3. Place a small drop of the gold chloride reagent on a glass slide.
4. Place the slide upside down over the well of the microwell slide.
5. The sample may be heated gently to assist in the transfer of a volatile unknown substance from the sample to the drop of reagent.
6. Observe and document the formation of any crystals that form. If necessary, stir gently.

2.3 Single Isomer Determination

If the direct or volatile method indicates that the substance is a single isomer, mix the unknown with an equal amount of either the known ***d-*** or ***l-*** isomer to determine which isomer is present.

3 Interpretations

Compare the shape/structure of crystals produced with crystals from known material and with reference to literature descriptions.

If the unknown is the ***d-*** isomer, there will be no change when mixed with the known ***d-*** isomer, but it will show the racemic mixture when mixed with the known ***l-*** isomer. If the

unknown is the ***l-*** isomer, there will be no change when mixed with the known ***l-*** isomer, but it will show the racemic mixture when mixed with ***d-*** isomer.

Indicates the presence of amphetamine (AuCl-A+) or methamphetamine (AuCl-M+) and can be used to determine the optical isomers present.

4 Literature and Supporting Documentation

Jay A. Siegel, "Forensic Identification of Controlled Substances," in *Forensic Science Handbook*, Volume II, ed. Richard Saferstein (Englewood Cliffs, New Jersey: Prentice-Hall, 1988), 80-81.

Charles C. Fulton, *Modern Microcrystal Tests for Drugs*, (John Wiley & Sons, Inc. 1969).

E. C. G. Clarke, "Microcrystal Tests," in *Isolation and Identification of Drugs*, ed. E. C. G. Clarke (London, William Clowes and Sons, 1969).

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Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5 Addition section 3 "Indicates the presence of amphetamine (AuCl-A+) or methamphetamine (AuCl-M+) and can be used to determine the optical isomers present."

MERCURIC IODIDE MICROCRYSTAL TEST

1 Reagents/Chemicals

- Mercuric iodide
- Concentrated HCl (37%)
- Purified H_2O

Mercuric Iodide Reagent: Add 27 ml concentrated HCl to 73 ml H_2O and mix well. Add sufficient mercuric iodide to the dilute HCl to obtain a saturated solution.

Storage: Keep tightly capped and store at room temperature.

Quality-test the reagent with heroin.

2 Procedure

1. Place a small drop of Mercuric Iodide reagent on a glass slide.
2. Add a small amount of the unknown to the reagent and let the unknown dissolve
3. Let stand for at least 5 minutes.
4. Record any observations.

3 Interpretation

Compare the shape/structure of crystals produced with crystals from known material and with reference to literature descriptions.

Generally used as a presumptive test for heroin (HgI+).

4 Literature and Supporting Documentation

Jay A. Siegel, “Forensic Identification of Controlled Substances,” in *Forensic Science Handbook*, Volume II, ed. Richard Saferstein (Englewood Cliffs, New Jersey: Prentice-Hall, 1988), 80-81.

Charles C. Fulton, *Modern Microcrystal Tests for Drugs*, (John Wiley & Sons, Inc. 1969)

E. C. G. Clarke, “Microcrystal Tests,” in *Isolation and Identification of Drugs*, ed. E. C. G. Clarke (London, William Clowes and Sons, 1969).

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

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Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5

AMMONIUM MOLYBDATE MICROCRYSTAL TEST

1 Reagents/Chemicals

- Ammonium molybdate
- Concentrated (70%) nitric acid
- Purified H_2O

Ammonium Molybdate Reagent: Prepare a saturated aqueous solution of ammonium molybdate.

Storage: Keep tightly capped and store at room temperature.

Quality-test with a small sample of red phosphorus.

2 Procedure

1. In the fume hood, place a 1 mg sample of the unknown into a test tube and add three drops of concentrated nitric acid.
2. Allow the reaction to sit in a fume hood for a 1-2 hours or until the brown fumes disappear.
3. Place a drop of the mixture on a glass slide and mix in a drop of saturated ammonium molybdate solution.
4. Let stand at least 3 minutes.
5. Record any observations.

3 Interpretations

Compare the shape/structure of crystals produced with crystals from known material and with reference to literature descriptions.

Generally used as a presumptive test for red phosphorus (P+).

4 Literature and Supporting Documentation

Donnell Christian, “Ammonium Molybdate Crystal Test for Phosphorus,” *Southwestern Association of Forensic Scientists Journal*, Volume 20, Issue 1, February, 1998, 23-25.

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

Concurrence

C. _Glen Johnson_ Date: 11/01/2002
Quality Assurance Coordinator

Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5

URANYL ACETATE MICROCRYSTAL TEST

1 Reagents/Chemicals

- Uranyl acetate
- Glacial acetic acid

Uranyl Acetate Reagent: Add glacial acetic acid to a saturated aqueous solution of Uranyl acetate until acidic (approximately 6% acid strength).

Storage: Keep tightly capped and store at room temperature.

Quality-test reagent with NaCl.

2 Procedure

1. Place a drop of Uranyl Acetate reagent on a glass slide.
2. Add a small amount of the unknown sample to the reagent.
3. Record any observations.

3 Interpretation

Compare the shape/structure of crystals produced with crystals from known material and with reference to literature descriptions.

Generally used as a presumptive test for sodium compounds (Na+).

4 Literature and Supporting Documentation

Jay A. Siegel, “Forensic Identification of Controlled Substances,” in *Forensic Science Handbook*, Volume II, ed. Richard Saferstein (Englewood Cliffs, New Jersey: Prentice-Hall, 1988), 80-81.

Charles C. Fulton, *Modern Microcrystal Tests for Drugs*, (John Wiley & Sons, Inc. 1969)

E. C. G. Clarke, “Microcrystal Tests,” in *Isolation and Identification of Drugs*, ed. E. C. G. Clarke (London, William Clowes and Sons, 1969).

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Hector Cadena Date: 11/01/2002
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Version #	Effective Date	Brief Description of Change(s)
00	12/01/2002	Original Issue, Minor Revision, Text from chapter 5

SILVER/COPPER NITRATE MICROCRYSTAL TEST

1 Reagents/Chemicals

- Silver nitrate
- Cupric nitrate
- Purified H_2O

Silver/copper nitrate reagent: Dissolve 0.1 g cupric nitrate and 0.1 g silver nitrate in 10 ml H_2O.

Storage: Keep tightly capped and store at room temperature.

Quality-test reagent with GHB.

2 Procedure

1. Place a drop of Silver/Copper Nitrate reagent on a glass slide.
2. Add a small amount of the unknown sample to the reagent.
3. Record any observations.

3 Interpretation

Compare the shape/structure of crystals produced with crystals from known material and with reference to literature descriptions.

Rectangular crystals occurring at the periphery of the exposed drop, indicates the presence of Gamma-Hydroxybutyrate (GHB+).

4 Literature and Supporting Documentation

Andera, K. M., H. K. Evans, and C. M. Wojcik. 2000. Microchemical Identification of Gamma-Hydroxybutyrate (GHB). *J. Forensic Science*, 45:665-668.

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

Concurrence

C. *Glen Johnson* Date: 11/01/2002
Quality Assurance Coordinator

Version #	Issue Date	Brief Description of Change(s)
00	12/01/2002	Original Issue

THIN-LAYER CHROMATOGRAPHY

1 Scope

To describe the use of thin-layer chromatography as an analytical method.

2 Related Documents

Marquis Reagent (CS-04-02)

pDMABA Reagent (CS-04-09)

3 Safety

1. 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.
2. 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 MSDS for the safe handling of the solvents and reagents used in this technique.
3. Developing solvents and indicator reagents should be discarded in an appropriate manner.

4 Equipment, Materials and Reagents

4.1 Equipment/Materials

1. Silica gel thin-layer chromatography plates
2. Developing chamber
3. Micropipettes (1-5 µL) or equivalent
4. UV light box (long and short wave)

4.2 Reagents

A. Approved TLC solvent systems

System ID	Solvent System (ratios of respective solvents)	Typical Drugs Analyzed
1.	50:25:15:10 (cyclohexane:toluene:acetone:diethylamine)	Marihuana
2.	18:1 (chloroform, sat. with ammonia:methanol)	LSD
3.	9:2 (chloroform:methanol)	LSD
4.	9:1 (acetone:chloroform sat. with ammonia)	LSD
5.	2:1:1 (n-butanol:acetic acid:water)	Psilocybin
6.	**T-1** 1.5:100 (ammonium hydroxide:methanol)	General
7.	**Davidow** 85:10:5 (ethyl acetate:methanol:ammonium hydroxide)	General
8.	13:1.9: 0.1 (methyl ethyl ketone: dimethylformamide: NH_4OH)	Mescaline

System ID	Solvent System (ratios of respective solvents)	Typical Drugs Analyzed
9	1:1 (ethyl acetate:hexanes)	Salvinorin A

B. Approved indicating reagents

Indicating Reagents	Typical Drugs Analyzed
Fast Blue RR	marihuana and THC
p-DMABA (CS-04-09)	LSD, psilocybin mushrooms, and indoles
Ninhydrin	mescaline and amines
Acidified Iodoplatinate	mescaline, opiates, and tertiary amines
Marquis (CS-04-02)	general substances
Potassium permanganate	general substances
Vanillin Reagent	Salvinorin A

C. Preparation of select indicating reagents

Fast Blue RR reagent:

- Purified H_2O, methanol, or ethanol
- Fast Blue RR salt

Dissolve 0.25 g Fast Blue RR salt in 50 mL solvent. Developed spot for THC appears red.

Ninhydrin reagent:

- Ninhydrin
- Acetone

Dissolve 0.5 g ninhydrin in 100 mL acetone. Developed spots appear red to purple.

Acidified Iodoplatinate reagent:

- 10% Platinic chloride solution
- 4% Potassium iodide solution
- Purified H_2O
- Concentrated (37%) HCl

Mix 5 mL 10% platinic chloride solution with 125 mL 4% KI solution. Dilute to 250 mL with purified water. Add 12.5 mL conc. HCl. Developed spots appear purple or blue.

Potassium permanganate reagent (1%):

- 0.5 *N* sulfuric acid
- Potassium permanganate ($KMnO_4$)

Add 1 g potassium permanganate to 100 mL 0.5 *N* sulfuric acid. Developed spots appear lighter than the background.

Vanillin Reagent:

- 50 mL ethanol
- 0.3 mL conc. sulfuric acid
- 1 g vanillin

Add 1 g vanillin to 50 mL ethanol, then add 0.3 mL conc. sulfuric acid. 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 standard will be analyzed on all plates. If the expected result of the standard is not obtained, the issue should be 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 positive determination is made when the spot(s) of the unknown substance matches the color and migration of the standard.

8 Limitations

1. TLC is not considered a confirmatory test and further analysis is necessary for the positive identification of a questioned substance.
2. 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

1. Relatively quick and easy technique
2. Can be used as a clean-up procedure for complex mixtures
3. Requires no expensive instrumentation

10 Literature and Supporting Documentation

J. M. Bobbitt, A. E. Schwarting, and R. J. Gritter. 1968. *Introduction to Chromatography*.

John A. Miller and E. F. Neuzil. 1979. *Organic Chemistry, Concepts and Applications*. D. C. Heath & Company, Lexington, Mass, 555.

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"Chromatographic Data, Thin Layer Chromatography Tables, Volume I, Sec. II.IV", *CRC Handbook of Chromatography*, Volume I, edited by Robert C. Weast, CRC Press, Division of the Chemical Rubber Company, 1972, 477-487.

"Practical Applications II.I Detection Reagents for Paper- and/or Thin Layer Chromatography", Volume 2, Section II, *CRC Handbook of Chromatography*, edited by Robert C. Weast, CRC Press, Division of the Chemical Rubber Company, 1972, 103-189.

E. Buel, C. N. Plum, and S. K. Frisbie. 1982. An Evaluation of a Partition Thin Layer Chromatography System for the Identification of Cannabinoids. *Microgram*, 15:145-157.

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R. B. Hughes and V. J. Warner, Jr., 1978. A Study of False Positives in the Chemical Identification of Marihuana. *J. Forensic Science*, 23:304-310.

Siebert DJ. Localization of Salvinorin A and Related Compounds in Glandular Trichomes of the Psychoactive Sage, Salvia divinorum. Annals of Botany. 93. 2004, pp. 763-771.

Preparer

Ruben A. Rendon, Jr. Date: 11/21/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoe M. Smith Date: 01/22/2009
Quality Assurance

Version #	Issue Date	Brief Description of Change(s)
	09/01/2001	Original Issue
01	12/01/2002	Minor Revision Modification of table of Approved TLC solvent systems Deletion Section 6 Calculation of Rf value
02	07/01/2003	Minor Revision with reference to Section 3.2 B test result deleted "usually"
03	10/10/2006	Addition to Section 4 "The results of the standard must be documented." Major Revision Section 4.2 regarding indicating reagents Modification to Section 4.1 #1 regarding the type of silica gel Modification to Section 4.1 #2 regarding developing chamber. Modification to Section 5 "…to test the system and ~~detection~~ indicating reagents. …" Modification to Section 6 #4 "…into a ~~solvent tray~~ developing chamber with…." Modification to Section 6 #8 "Compare the ~~location~~ migration of the sample spot to that of the standard." Modification Section 7 "…the color and ~~location~~ migration of the standard."
04	02/09/2009	Major revision – Sections 4.2 and 10 Advisory Board 11/20/2008

ULTRAVIOLET/VISIBLE SPECTROPHOTOMETRY (UV/VIS)

1 Scope

A nondestructive technique for the preliminary identification of controlled substances, dangerous drugs and other substances.

2 Safety

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

3 Equipment, Materials and Reagents

1. Double-beam UV/visible spectrophotometer
2. Quartz cuvettes, matched pair or equivalent
3. An appropriate solution for the sample.
   a) *Acidic solutions, such as 0.2* N H_2SO_4 *or 0.1* N *HCl*
   b) *Basic solutions, such as concentrated NaOH or 1.0* M Na_2CO_3
   c) *Methanol or ethanol*

4 Standards, Controls and Calibrations

1. Quarterly UV/VIS instrument performance verification check
2. For comparison purposes, refer to reliable published reference material, analyze known control samples or refer to an in-house spectral collection produced from known samples.
3. Appropriate blanks should be analyzed as deemed necessary by the chemist.

5 Procedure

5.1 Spectrophotometer Operating Conditions

1. The wavelength range used for the UV/VIS analysis of most drug samples is 340 to 220 nm.
2. 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, while 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. Each spectrum will be printed, labeled with laboratory case number, exhibit number, date, examiner's handwritten initials and method of sample preparation (if not listed on the worksheet), and retained in the case file.

6 Interpretation

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

B. Mark the worksheet in the column under the UV heading that matches the solvent used for the test; or if other than aqueous acid or base is used, document the test and solvent in the "Other Tests" column of the worksheet.

1. A checkmark (√) on the worksheet may be used to indicate that the test was performed.
2. A plus sign (+) on the worksheet may be used to indicate that the sample spectrum supports the identification of the reported substance or substance class.
3. If the sample spectrum is interpreted as a positive test, document the source of any reference spectrum employed. Use the approved list of reference abbreviations if applicable; if some other reference is used, provide a complete citation.
4. The interpretation of a sample's spectrum may be reflected directly on the spectrum itself.

7 Limitations

1. An ultraviolet spectrum is not specific, and a positive identification cannot be made exclusively on the basis of UV/VIS analysis.
2. 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.
3. 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

1. The test is quick and easy to perform.
2. Usually very little sample preparation is required.

3. UV/VIS analysis is a good screening tool and routine analysis may provide information regarding the general concentration of the sample (strong, average or weak) and the presence or absence of some dilutants and adulterants.

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

5. May provide a quick and easy quantitation of some drugs/dilutants.

9 Literature and Supporting Documentation

Douglas A. Skoog and Donald M. West, *Principles of Instrumental Analysis* (New York: Holt, Rinhart, and Winston, Inc., 1971).

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Kenneth A. Rubinson, *Chemical Analysis*, ISBN 0-316-76087-0.

Donald L. Pavia, Gary M. Lampman, George S. Kriz Jr., *Introduction to Spectroscopy*, ISBN 0-7216-7119-5.

Robert M. Silverstein, G. Clayton Bassler, Terence C. Morill, *Spectrometric Identification of Organic Compounds*, 4th edition, ISBN 0-471-02990-4.

S. Sternhell and J. R. Kalman, *Organic Structures from Spectra*, ISBN 0-471-90647-6.

Preparer

Larry Todsen Date: 06/21/2007
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 06/21/2007
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
	05/15/2002	Section 7.1, 5.3 #5 Added to record interpretations Section 7.1, 6 Edited spectral comparisons and added where to document interpretations.
01	12/01/2002	Minor Revision Modification Section 4 change calibration to performance verification Modification Section 5.3, 4 "Each spectrum will be printed, labeled with laboratory case number, exhibit number, date, examiner's handwritten initials and method of sample preparation (if not listed on the worksheet), and retained in the case file. Deletion Section 5.3 #5 Record Interpretations Modification Section 6 "A checkmark (√) on the worksheet indicates that the test was performed. The interpretation of the spectra may be reflected directly on the spectrum or by the presence of a plus sign (+) on the worksheet below the UV category which is inferred to indicate that the item supports the identification of the reported substance or class of substances
02	07/01/2003	Minor revision with reference to UV/VIS Deletion Section 5.3 3. with reference to second spectrum
03	07/20/2007	Addition Section 3, #3, b: "…concentrated NaOH or" Addition Section 6: A. Evaluate the sample's UV spectrum by comparing it to reference literature or to one or more UV spectra derived from known samples. B. Mark the worksheet in the column under the UV heading that matches the solvent used for the test; or if other than aqueous acid or base is used, document the test and solvent in the "Other Tests" column of the worksheet. 1. A checkmark (√) on the worksheet may be used to indicate that the test was performed. 2. A plus sign (+) on the worksheet may be used to indicate that the sample spectrum supports the identification of the

03	07/20/2007	reported substance or substance class. 3. If the sample spectrum is interpreted as a positive test, document the source of any reference spectrum employed. Use the approved list of reference abbreviations if applicable; if some other reference is used, provide a complete citation. 4. The interpretation of a sample's spectrum may be reflected directly on the spectrum itself. Deletion Section 6: ~~The spectra obtained are evaluated with reference to documented sources or spectra from known samples.~~ ~~A checkmark (√) on the worksheet indicates that the test was performed. The interpretation of the spectra may be reflected directly on the spectrum or by the presence of a plus sign (+) on the worksheet below the UV/VIS category which is inferred to indicate that the item supports the identification of the reported substance or class of substances.~~

FOURIER TRANSFORM INFRARED (FTIR) SPECTROPHOTOMETRY

1 Scope

A non-destructive analytical technique used for the characterization and identification of suspected controlled substances, dangerous drugs and other substances.

2 Safety

Use appropriate safety equipment when preparing reagents. Refer to the MSDS for additional safety information for specific chemicals.

3 Equipment, Materials and Reagents

1. Fourier transform infrared spectrophotometer
2. Agate mortar and pestle
3. Hydraulic press and KBr die, or hand press
4. Potassium bromide (KBr), dry
5. NaCl or KBr windows (*e.g.*, 2mm x 13 mm)
6. Nujol
7. Laboratory oven
8. Special cells for liquids or vapors

4 Standards, Controls, and Calibration

1. Quarterly performance verification check.
2. The spectrum obtained is compared to the appropriate reference material or spectra from known samples.

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) Volatiles 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 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.

5.2 Sample Analysis

1. Collect and print spectra with a resolution of at least 4 cm^{-1} from 4000 cm^{-1} to 400 cm^{-1} (or 600 cm^{-1} with NaCl) versus % transmittance (0-100). Spectral peaks should be of sufficient intensity to make an accurate comparison to known reference standards or published spectral data.

2. Spectra will be labeled with laboratory case number, exhibit number, date, examiner's handwritten initials, and method of sample preparation (if not shown on the worksheet) and retained in the case file. Instrument operating conditions will be retained in the case folder or available in a retrievable format.

3. Document the confirmation of the unknown spectra to a known reference and indicate the source of the reference in the case file (published or otherwise lab generated).

6 Interpretation

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

Library searches can be used to provide useful information pertaining to the identity of a compound, but should not be used as a replacement for verifying positive identification, due to the abridged nature of the spectra found in search libraries. Results from library searches need not be printed.

For confirmation, the unknown spectrum must match spectra from known standards or libraries.

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

7 Limitations

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

2. 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.

3. Infrared cannot usually be used to distinguish optical isomers.

8 Advantages

1. Infrared is specific for the identification of controlled substances, dangerous drugs, and dilutants and can be used as a confirmatory test.

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

3. 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 and Supporting Documentation

"Standard Practice for Describing and Measuring Performance of Fourier Transform Infrared (FT-IR) Spectrophotometers: Level Zero and Level One Tests," ASTM E 1421-91, 1991.

A. F. Fell, *Clarke's Isolation and Identification of Drugs*, (London: The Pharmaceutical Society of Great Britain, 1986).

Forensic Science Handbook, Volume III, ed. by Richard Saferstein, (Englewood Cliffs, N. J.: Regents/Prentice Hall, 1993).

Douglas A. Skoog, *Principles of Instrumental Analysis*, 3rd Edition, (New York: Saunders College Publishing, 1985) 148-149.

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

Concurrence

C. *Glen Johnson* Date: 11/01/2002
Quality Assurance Coordinator

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
	05/15/2002	Section 7.2, 5.2 #3 Moved from Interpretations and modified to require documentation of the comparison of questioned spectra to those of known references and indicate the source used. Section 7.2, 6 Edited interpretations and added library lists and how documented.
01	12/01/2002	Minor Revision Modification Section 4, 1 change calibration to performance verification. Modification Section 5.2, 3 Document the confirmation of the unknown spectra to a known reference and indicate the source of the reference in the case file (published or otherwise lab generated).

GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)

1 Scope

An analytical technique for the characterization and identification of suspected controlled substances, dangerous drugs and other substances.

2 Safety

1. Use appropriate safety equipment when preparing reagents and handling volatile chemicals. Refer to the MSDS for additional safety information for specific chemicals.
2. Properly secure high-pressure gas cylinders.
3. Use caution around hot surfaces such as oven interiors and injection and detector ports.

3 Equipment, Materials and Reagents

1. Gas chromatograph/mass spectrometer analytical instrument
2. Helium
3. Auto-sampler vials and caps (where applicable)
4. Microliter syringe (where applicable)

4 Standards, Controls and Calibration

1. Calibration of the mass spectrometer is accomplished by tuning the instrument to ensure that the mass-to-charge ratios (m/z) are assigned correctly and to provide leak detection.

   a) *The instrument should be tuned according to the manufacturer's specifications and may be tuned more frequently as deemed necessary by the analyst and/or the laboratory supervisor.*

   b) *Maintain records of the tune in a file in the laboratory. If the tune is not successful, the instrument should be taken out of service until corrective action is taken.*

2. Periodically the analyst should inject control blanks, consisting of the same solvent used in sample preparation, to verify that the column, solvent and laboratory glassware used are clean prior to the analysis of evidence samples.
3. For exhibits that contain an insufficient amount of material for two independent samples, a method blank must be prepared using the same parameters as the evidence sample and analyzed before the evidence sample. Maintain the resulting chromatograms in the case folder or in a retrievable form.

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 a reasonable time for unknown or unexpected compounds to elute.

2. Print and retain the program parameters in the case folder or in a retrievable form.

5.2 Sample Preparation and Analysis

1. Extract samples into a suitable solvent before they are injected into the instrument. As a general rule, avoid methanol and other highly polar solvents that can cause degradation of the GC column lining.

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 evaluated on the primary TIC.

   e) Label each printout with the laboratory case number, exhibit number, date, examiner's handwritten initials, and if not shown on the worksheet, the method of sample preparation.

4. Document the comparison of the spectrum of each reported substance to a known reference spectrum, and indicate the source of the reference (published or otherwise lab-generated).

5.3 Retention Time Analysis

1. Select the appropriate drug reference standard for comparison with the unknown substance. Ensure that the compounds are in the same chemical form, for example both in the base or salt form.

2. Analyze the prepared drug reference standard and prepared unknown sample(s) and compare the retention time of the peaks. The drug reference standard must be run in conjunction with the samples.

3. Print and retain the chromatograms of all relevant samples, blanks, and standards in the case folder. The chromatograms will be labeled with laboratory case number, exhibit number, date, examiner's handwritten initials, and method

of sample preparation (if not shown on the worksheet) and retained in the case file. Instrument operating conditions will be retained in the case folder or available in a retrievable format.

6 Interpretation

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

B. Library searches can be used to provide useful information pertaining to the identity of a compound, but should not be used as a replacement for verifying positive identification, due to the abridged nature of the spectra found in search libraries. Results from library searches need not be printed.

C. The difference between retention times of the known and unknown samples must be less than three percent.

$$PercentDifference = \frac{\left| retention_{std} - retention_{unk} \right|}{retention_{std}} \times 100$$

7 Limitations

1. When analysis by GC/MS is unable to provide positive identification in some instances, another technique (FTIR, derivatization, etc.) must be utilized to provide positive identification For example, certain stereo- and geometric isomers give identical or very similar results.

2. 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.

3. It may be difficult to identify individual compounds in a homologous series.

8 Advantages

1. Generally, mass spectra of controlled substances are specific to single compounds and may be used for identification.

2. It may be possible to separate and identify complex mixtures that are difficult to separate through ordinary clean-up procedures.

3. The technique is useful for analyzing small sample amounts that may be difficult to identify using other techniques.

4. An autosampler, which increases the efficiency of analysis of numerous samples and functions unattended, may be attached to the GC/MS.

9 Literature and Supporting Documentation

Douglas A. Skoog, *Principles of Instrumental Analysis*, 3rd Edition, (New York: Saunders College Publishing, 1985) 523-535, 554.

F. W. McLafferty, *Interpretation of Mass Spectra*, 3rd Edition, (Mill Valley, California: University Science Books, 1980).

J. Throck Watson, *Introduction to Mass Spectroscopy: Biomedical, Environmental, and Forensic Applications*, (New York: Raven Press Books, 1140 Avenue of the Americas, 1976).

Preparer

Ruben A. Rendon, Jr. Date: 03/03/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 04/08/2008
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
	05/15/2002	Section 7.3, 5.2 #3 Moved from interpretations and modified to require documentation of the comparison of questioned spectra to those of known references and indicate the source used.
01	12/01/2002	Minor Revision Modification Section 5.2, 3 "Document the comparison of the unknown spectra to a known reference and indicate the source of the reference in the case file (published or otherwise lab-generated)."
02	07/01/2003	Modification Section 4, 3 "A method blank will be run for samples that were completely consumed by analysis."
03	10/10/2006	Addition Section 3 #2 "Helium" Modification to Section 4 #3 "For exhibits that contain an insufficient amount of material for two independent samples, a method blank must be prepared using the same parameters as the evidence sample and analyzed before the evidence sample. ~~A method blank will be run for samples that were completely consumed by analysis. If contamination is indicated, the problem must be resolved before the analysis is repeated.~~"

Version #	Effective Date	Brief Description of Change(s)
04	07/20/2007	Modification Section 5.2: 2. Review and identify the major peaks in the GC/MS total ion chromatogram (TIC). ~~Print and retain the charts depicting~~Document the results of the GC/MS analysis by printing relevant spectra and retaining them in the case file. Include the following: a) The complete Total Ion Chromatogram (TIC) b) Mass spectra for all significant peaks as determined by the analyst.~~corresponding to controlled substances and/or other substances reported~~ c) Mass spectra for any other peaks examined that are deemed relevant by the analyst d) Label each printout with the laboratory case number, exhibit number, date, examiner's handwritten initials, and if not shown on the worksheet, the method of sample preparation ~~(if not shown on the worksheet)~~. 3. Document the comparison of the spectrum of each reported substance ~~unknown spectra~~ to a known reference spectrum, and indicate the source of the reference in the case file (published or otherwise lab-generated).
05	05/05/2008	Major revision – Sections 5.2, 5.3 and 6 Advisory Board 02/27/2008

GAS CHROMATOGRAPHY (GC)

1 Scope

An analytical technique for the characterization and identification of suspected controlled substances, dangerous drugs and other substances.

2 Safety

1. Use appropriate safety equipment when preparing reagents and handling volatile chemicals. Refer to the MSDS for additional safety information for specific chemicals.
2. Properly secure high-pressure gas cylinders.
3. Use caution around hot surfaces.

3 Equipment, Materials and Reagents

1. Gas chromatograph equipped with a flame ionization detector (FID)
2. Appropriate carrier and fuel gases

4 Standards, Controls and Calibrations

Appropriate drug reference standards must be analyzed on the instrument used for comparison of retention time.

5 Procedure

1. Select the appropriate drug reference standard for comparison with the unknown substance. Ensure that the compounds are in the same chemical form, for example both in the base or salt form.
2. Analyze the prepared drug reference standard and prepared unknown sample(s) and compare the retention time of the peaks. The drug reference standard must be run in conjunction with the samples.
3. Print and retain the chromatograms of all relevant samples, blanks, and standards in the case folder. The chromatograms will be labeled with laboratory case number, exhibit number, date, examiner's handwritten initials, and method of sample preparation (if not shown on the worksheet) and retained in the case file. Instrument operating conditions will be retained in the case folder or available in a retrievable format.

6 Interpretation

1. Examine the resulting chromatogram to determine if the separation between components is adequate for the intended purpose.
2. The difference between retention times of the known and unknown samples must be less than three percent.

$$PercentDifference = \frac{\left| retention_{std} - retention_{unk} \right|}{retention_{std}} \times 100$$

7 Limitations

1. Two or more compounds, especially those similar in chemical structure, can have the same retention time under identical GC conditions.
2. Co-eluting compounds may mask the peak of interest.
3. The elevated injection port temperature can result in partial decomposition of certain compounds.

8 Advantages

1. Excellent technique for separating chemical components. Allows quantitation of complex mixtures.
2. Relatively simple sample preparation.
3. Many different compounds can be analyzed on the same instrument by varying the GC conditions, such as the carrier gas flow rate and the oven temperature program.

9 Literature and Supporting Documentation

D.A. Skoog and J.J. Leary, *Principles of Instrumental Analysis*, Saunders College Publishing, 1992, pp. 432, 434, 622.

Modern Practice of Gas Chromatography, edited by Robert L. Grob (New York: John Wiley and Sons, 1977) 153-211 and 611-613.

HP5890A Gas Chromatograph Shelf Reference Manual, Volume I and Volume II, 1983-1986, Hewlett Packard Co., Route 41, Avondale, Penn. 19311.

Preparer

Ruben A. Rendon, Jr. Date: 03/03/2008
Controlled Substance Advisory Board Chair

Concurrence

Zoë M. Smith Date: 04/08/2008
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
01	12/01/2002	Minor Revision
02	10/10/2006	Addition Section 3 #2 “Appropriate Carrier and Fuel Gases“
03	05/05/2008	Major revision – Sections 3, 4, 5, and 6 Minor revision – Sections 7 and 8 Advisory Board 02/27/2008

POLARIMETRY

1 Scope

To determine the direction of rotation (*dextrorotatory* or *levorotatory)* of optically active controlled substances, dangerous drugs and other substances.

2 Safety

When using this instrument, be sure to use the appropriate personal protective equipment.

3 Equipment, Materials and Reagents

1. Polarimeter
2. 100 mm and/or 200 mm observation tube(s)
3. Centrifuge
4. Filter paper
5. An optically inactive solvent (such as methanol)

4 Standards, Controls and Calibration

1. Proper functioning of the instrument should be checked by verifying the direction of optical rotation of a known standard (e.g. l-ephedrine) prior to each set of samples. Document the results of the check.

2. Perform zero-setting for calibration each time the instrument is turned on for measurement.

5 Procedure

5.1 Zero Calibration of Instrument

1. Allow instrument to warm up prior to use.

2. Carefully fill the same observation tube used for the sample and with the same optically inactive solvent used to prepare the sample. Make sure there are no trapped air bubbles.

3. With the observation tube in place, set the instrument to read zero and proceed to sample analysis.

5.2 Sample Preparation and Analysis

1. Extract the compound of interest with an optically inactive solvent.

2. If the solution is cloudy, filter and/or centrifuge the sample, until clear. Prepare enough solution to completely fill the appropriate observation tube.

3. Completely fill the observation tube with the sample solution.

4. Place the tube into the instrument.

5. Record the direction of rotation of the unknown sample.

6 Interpretation

1. A positive value indicates a “d” (dextrorotatory) rotation.
2. A negative value indicates a “l” (levorotatory) rotation.
3. A null value indicates an optically inactive sample.

7 Limitations

1. The sample must be clean to get quality results; contaminated samples can give false results.
2. A racemic mixture, which contains equal amounts of both “*d*” and “*l*” isomers, will appear to be optically inactive.

8 Advantages

1. Determining the optical rotation of a controlled substance, especially amphetamine or methamphetamine, can provide information relative to the manufacturing process.
2. Allows the identification of *propoxyphene* as *dextropropoxyphene* as listed in the *Texas Drug Laws* in the Health and Safety Code.

9 Literature and Supporting Documentation

Michael Bass, *Handbook of Optics Devices, Measurements, and Properties* (McGraw-Hill, Inc., 1995), 22.3-22.7.

Bud Warner, *Instrument of Science: An Historical Encyclopedia* (Garland Publishing, Inc., 1998), 473.

A Dictionary of Chemistry, 3rd Edition, edited by John Daintith (Oxford: Oxford University Press, 1996), 390-391 and 352-353.

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

Concurrence

C. *Glen Johnson* Date: 11/01/2002
Quality Assurance Coordinator

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
01	12/01/2002	Minor Revision

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

1 Scope

An analytical technique for the characterization and identification of suspected controlled substances, dangerous drugs, and other substances.

2 Safety

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

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

3 Equipment, Materials, and Reagents

1. High performance liquid chromatograph system including solvent degasser, pump system, injector, and detector such as photodiode array detector.

2. Microliter syringe (with special tip to match injector valve).

3. Equipment to prepare elution solvents such as balances, graduated cylinders, volumetric flasks, pipets, pH meter, and magnetic stirrer.

4. Vacuum filtration apparatus for solvent filtration with 0.45 µm membrane filter.

5. Syringe filter for sample filtration through 0.45 µm membrane filter.

6. High purity solvents (HPLC grade or better) such as water, methanol, and acetonitrile. It is not normally necessary to filter HPLC grade solvents before use.

7. Distilled water (solutions made with distilled water must be filtered before use).

4 Standards, Controls, and Calibration

A. Appropriate drug reference standards must be injected to compare the retention time and UV spectrum of the unknown substance to a known sample in the solvent system used.

B. The photodiode array detector should be calibrated periodically as described in the instrument manual.

5 Procedure

1. Select the appropriate drug reference standard for comparison with the unknown substance. Prepare the drug reference standard and the evidence sample using the same solvent and/or extraction procedure. 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 folder. The chromatograms and spectra will be labeled with laboratory case number, exhibit number (where applicable), date, examiner's handwritten initials, and method of sample preparation (if not shown on the worksheet). As a minimum, the column type and mobile phase should be included in the case folder with the method information.

6 Interpretation

A. Examine the resulting chromatograms to determine if the retention times and UV spectra of the unknown and standard match.

B. If quantitation is to be performed in the system used, examine the chromatogram of the sample to determine if the compound of interest is well resolved from peaks due to impurities.

7 Limitations

A. Two or more compounds, especially those similar in chemical structure, can have similar retention times under the same chromatographic conditions.

B. If component peaks are not well resolved, it might be necessary to change the method in order to obtain useful UV spectra.

C. Certain solvents can cause decomposition of some samples, such as the hydrolysis of heroin.

D. HPLC retention times and UV absorbance spectra are not considered confirmatory tests and further analysis is necessary for the positive identification of an unknown substance.

E. Not all substances absorb UV or visible light, and therefore, these compounds would not be detected with a photodiode array detector. Many other detectors are available for HPLC which could be used in these circumstances, as could derivatization techniques which add a chromophore to compound.

F. The absorbance of many compounds depends on the solvent used which makes using standard libraries of UV spectra to be used to determine an unknown compound more difficult.

8 Advantages

A. Excellent technique for separating chemical components. Allows quantitation of complex mixtures.

B. Better than gas chromatography in separating analytes that are thermally unstable or not readily volatile.

C. Usually, relatively simple sample preparation.

D. Many different compounds can be analyzed on the same instrument and column by varying the elution solvent system.

E. Adjusting the monitoring wavelength allows the UV absorbance peak to be used which results in the maximum sensitivity and linear range.

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

9 9 Literature and Supporting Documentation

T. Kupiec, M. Slawson, F. Pragst, and M. Herzler, in Clarke's Analysis of Drugs and Poisons, 3rd ed.; A. C. Moffat, M. D. Osselton, and B. Widdop, Eds.; Pharmaceutical Press, Chicago, 2004; pp. 500-534.

D. Northrop, in Forensic Science Handbook, 2nd ed.; R. Saferstein, Ed.; Prentice-Hall, Upper Saddle River, New Jersey, 2002; pp.41-116.

D. A. Skoog, D. M. West, and F. J. Holler, Fundamentals of Analytical Chemistry, 5th ed.; W. B. Saunders, San Francisco, 1988; pp.644-666.

H. H. Willard, L. L. Merritt, Jr., J. A. Dean, and F. A. Settle, Jr., Instrumental Methods of Analysis, 7th ed.; Wadsworth, Belmont, California, 1988; pp.614-655.

Preparer

Andrew Macey Date: 04/04/2005
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 04/04/2005
Quality Assurance Specialist

Version #	Effective Date	Brief Description of Change(s)
00	05/01/2005	Original Issue

QUANTITATION BY ULTRAVIOLET SPECTROPHOTOMETRY

1 Scope

To establish a procedure to determine the concentration of a controlled substance, dangerous drug, or other substance in a sample using ultraviolet spectrophotometry.

2 Safety

Refer to Standard Operating Procedure 7.1 for general operation.

3 Equipment, Materials and Reagents

1. Double-beam UV/Visible spectrophotometer
2. Quartz cuvettes, matched pair or equivalent
3. Analytical balance

4 Standards, Controls and Calibrations

1. Quarterly performance verification
2. Reference solvent blank
3. $E_{1cm}^{0.1\%}$ (*E*-Value) for the compound of interest (This may be obtained from reference literature or determined/confirmed with laboratory standards on the instrument prior to using this technique for quantitation. If the latter procedure is used, the results will be documented in retrievable format.)
4. Controlled substance reference standard for the drug to be quantitated
5. Each instrument shall have a valid three-point linearity plot using any UV-absorbing substance. If major instrument repairs (*e.g.,* replacement of the source or detector) are performed, the linearity will be re-confirmed.

5 Procedure

5.1 Operating Conditions

1. The wavelength range used for the UV analysis of most drug samples is 340 to 220 nm.
2. The range may be expanded to accommodate certain substances, such as alkyl nitrites and GHB.

5.2 Sample Preparation

1. Obtain a representative sample of the substance requiring quantitation. The amount needed will vary according to the concentration of the controlled substance in the sample and the *E*-value or absorptivity of the controlled substance. For best results:

a) *Adjust the concentration of the controlled substance so that the absorbance is strong enough to differentiate the peaks from background noise and yet weak enough to remain in the linear absorbance range.*

b) *The sample must be diluted such that the absorbance is within the determined linear range.*

2. For powdered (solid) samples:

a) *To reduce the effects of the inherent percent error in weighing the sample on the final quantitation results, the analyst should use a larger quantity of the sample and dilute as necessary to obtain a solution that gives an absorbance in the linear range.*

b) *Some samples may require extraction before they can be quantitated.*

3. For liquid samples:

a) *Liquid samples may vary greatly in concentration and should be extracted before quantitation, using an appropriate procedure for the substance being analyzed.*

5.3 Sample Analysis

1. A baseline spectrum should be obtained by scanning the desired wavelength range with a blank solution in both the reference and sample cuvettes. If necessary on instruments without auto-zero features, adjust the baseline to zero. The baseline need not be retained in the case file, unless desired by analyst.

2. Collect a spectrum of the sample.

3. Calculations to determine the concentration should be included in the case folder, either on the UV chart or on a separate page.

4. Each spectrum will be printed, labeled with laboratory case number, exhibit number, date, examiner's handwritten initials and method of sample preparation (if not listed on the worksheet), and retained in the case file.

6 Interpretation

1. The concentration will be calculated by application of the Beer/Lambert Law:

A = abc, where

A = absorbance value

ab = $E_{1cm}^{0.1\%}$ value [b = path length = 1 cm]

c = concentration

2. Note that the *E*-values in *Clarke's* are at 1.0% and must be divided by 10 in order for the resultant calculation to yield a concentration (c) value of mg/ml (0.1 %).

3. For basic drugs, report the quantitation results in base form. The concentration as the salt may be reported only if the analyst has identified the salt form by an accepted analytical procedure.

7 Limitations

1. UV quantitation is not suitable for samples that do not absorb UV light or for those that contain interfering compounds (such as nicotinamide and pseudoephedrine in methamphetamine samples) that modify the absorbance of the sample at the quantitation wavelength.

2. This technique is usually not suitable for samples with more than one controlled substance.

3. Samples that are not suitable for ultraviolet quantitation may be quantitated using an alternate technique such as gas chromatography.

8 Advantages

When analyzing relatively pure compounds, the test is quick and easy to perform, and requires less time and sample preparation than quantitation using gas chromatography.

9 Literature and Supporting Documentation

Refer to the instrumental analysis section for ultraviolet spectrophotometry.

Preparer

Hector Cadena Date: 11/01/2002
Controlled Substance Advisory Board Chair

Concurrence

C. _Glen Johnson_ Date: 11/01/2002
Quality Assurance Coordinator

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
	05/15/2002	Section 8.1, 4 Modified source of E-Value, Added requirement of a valid linearity plot for the instrument.
01	12/01/2002	Minor Revision Modification Section 4, 1 change calibration to performance verification. Deletion Section 5.2, 1 b Addition Section 5.2, 1 b "The sample must be diluted such that the absorbance is within the determined linear range."

QUANTITATION BY GAS CHROMATOGRAPHY WITH INTERNAL STANDARD

1 Scope

To establish a procedure to determine the concentration of a controlled substance, dangerous drug, or other substance in a sample using gas chromatography with an internal standard.

2 Safety

Refer to the GC procedure for general operation.

3 Equipment, Materials and Reagents

1. Gas chromatograph
2. Appropriate carrier and fuel gases
3. Analytical balance
4. Suitable solvents for sample preparation
5. Controlled substance reference standard for the drug to be quantitated
6. Internal standards such as n-C-14, n-C-24 or n-C-28 hydrocarbons
7. Injection syringe

4 Standards, Controls and Calibrations

1. Each instrument shall have a valid three point linearity plot using a common controlled substance (*e.g.,* cocaine). If major instrument repairs (*e.g.,* replacement of the detector) are performed, the linearity will be re-confirmed.

2. 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 the following:

   n-C-14 for amphetamine and methamphetamine

   n-C-24 for cocaine

   n-C-28 for opiates

   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. If a fresh internal standard is prepared and used for any of the quantitation samples, then all samples must be prepared using the new solution.

3. Prepare a known standard using the reference sample of the controlled substance to be quantitated.

4. Reanalysis of the known standard is performed to verify that the response is reproducible (within 5% of the expected amount). If the response does not meet

the expected value, then the issue will be resolved prior to use on casework samples.

5 Procedures

5.1 Preparation

1. Prepare the sample to be quantitated in the same manner as the known reference standard. Document the amount of sample and internal standard solution used.

5.2 Analysis

1. Instrument operating conditions will be retained in the case folder or available in a retrievable format.
2. Analyze the prepared known standard and prepared unknown sample.
3. The chromatograms of all relevant samples and standards will be printed, labeled with laboratory case number, exhibit number, date, examiner's handwritten initials, and method of sample preparation (if not listed on the worksheet), and retained in the case file.
4. Determine the concentration of the sample, expressed either as a percentage or as weight/volume.

6 Interpretation

The concentration of the drug present is proportional to the ratio of the unknown and standard integrated areas with respect to the integrated areas of the internal standard of each.

$$Conc_{unk} = \frac{Area_{unk} / Area_{IS}}{Area_{std} / Area_{IS}} \times Conc_{std}$$

7 Limitations

1. Standards of known purity must be used.
2. The peak to be quantitated must be a single component peak and completely resolved.

8 Advantages

1. Samples containing complex mixtures can be quantitated.
2. Many different compounds can be analyzed on the same instrument and column by varying the GC conditions.

9 Literature and Supporting Documentation

D. A. Skoog and J. J. Leary, *Principles of Instrumental Analysis*, (Saunders College Publishing, 1992), 432, 434, 622.

Preparer

Larry Todsen Date: 06/12/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 06/12/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
	09/01/2001	Original Issue
	05/15/2002	Section 8.2, 4 Added requirement of a valid linearity plot for the instrument.
01	12/01/2002	Major Revision and Removal of Quantitation by GC with an External Standard Addition Section 4, 4 "Reanalysis of the known standard is performed to verify that the response is reproducible (within 5% of the expected amount). If the response does not meet the expected value, then the issue will be resolved prior to use on casework samples."
02	12/01/2003	Deletion to Section 3 #1 specifications for detector
03	10/10/2006	Addition Section 3 #2 "Appropriate carrier and fuel gases"

QUANTITATION BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

1 Scope

To establish a procedure to determine the amount or concentration of a controlled substance, dangerous drug, or other substance in a sample using high performance liquid chromatography.

2 Safety

Refer to the HPLC procedure for general operation.

3 Equipment, Materials, and Reagents

1. High performance liquid chromatography system.
2. Analytical balance.
3. High purity solvents (HPLC grade or better) such as water, methanol, and acetonitrile. It is not normally necessary to filter HPLC grade solvents before use.
4. Assayed controlled substance reference standard for the drug to be quantitated.
5. Microliter syringe (with special tip to match injector valve) of sufficient volume to completely flush sample loop of injector.
6. Equipment to prepare elution solvents such as balances, graduated cylinders, volumetric flasks, pipets, pH meter, and magnetic stirrer.
7. Vacuum filtration apparatus for solvent filtration with 0.45 µm membrane filter.
8. Distilled, not deionized, water (solutions made with distilled water must be filtered before use.

4 Standards, Controls, and Calibration

A. Each controlled substance shall have a calibration curve for the elution solvent to be used, consisting of several different concentrations, to establish both linearity as well as calibration.

B. Prepare a known standard using the assayed reference sample of the controlled substance.

C. Reanalysis of the known standard is performed to verify that the response in reproducible (within 5% of the expected amount). If the response does not meet the expected value, then the issue will be resolved prior to use on casework samples. This will normally be accomplished by constructing a new calibration curve.

5 Procedures

1. Prepare the sample to be quantitated in the same manner as the known reference standard. It is desirable for both the sample and standard to be dissolved in the elution solvent before injection. Document the amount of sample and volume of solvent used.

2. Instrumental parameters will be retained in the case folder.

3. Analyze the prepared known standard and prepared unknown sample. While it is not necessary to prepare multiple solutions of the standard or sample, it is desirable to obtain at least two runs from each solution. The duplicate standard runs will indicate stability of the instrument. The value reported for the sample should be taken from the average of at least two runs.

4. The chromatograms of all relevant standards and samples will be printed, labeled with the laboratory case number, exhibit number (where applicable), date, examiner's handwritten initials, and method of sample preparation (if not listed on the worksheet), and retained in the case file. As a minimum, the column type and mobile phase should be included in the case folder with the method information.

5. Determine the concentration of the sample in the analyzed solution, and from this value and the solution volume and sample weight, determine either the percentage or concentration expressed as weight/volume of controlled substance in the sample.

6 Interpretation

The concentration of the drug present in the unknown solution is determined from the equation given by the linear regression of the calibration curve. The peak area of the unknown should fall in the proven linear range of the calibration curve.

7 Limitations

A. Standards of known purity must be used.

B. The peak to be quantitated must be free from other substances which absorb at the wavelength used for analysis.

C. Those samples which do not absorb in the UV-Visible region can not be analyzed using a photodiode array detector.

8 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 elution solvent.

9 Literature and Supporting Documentation

T. Kupiec, M. Slawson, F. Pragst, and M. Herzler, in Clarke's Analysis of Drugs and Poisons, 3rd ed.; A. C. Moffat, M. D. Osselton, and B. Widdop, Eds.; Pharmaceutical Press, Chicago, 2004; pp. 500-534.

D. Northrop, in Forensic Science Handbook, 2nd ed.; R. Saferstein, Ed.; Prentice-Hall, Upper Saddle River, New Jersey, 2002; pp.4l-ll6.

D. A. Skoog, D. M. West, and F. J. Holler, Fundamentals of Analytical Chemistry, 5th ed.; W. B. Saunders, San Francisco, 1988; pp.644-666.

H. H. Willard, L. L. Merritt, Jr., J. A. Dean, and F. A. Settle, Jr., Instrumental Methods of Analysis, 7th ed.; Wadsworth, Belmont, California, 1988; pp.614-655.

Preparer

Andrew Macey Date: 04/04/2005
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 04/04/2005
Quality Assurance Specialist

Version #	Effective Date	Brief Description of Change(s)
00	05/01/2005	Original Issue

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 Documents

CS-09-02 Modified Molisch Test

CS-07-02 Fourier Transform Infrared Spectrophotometry

3 Safety

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.

Refer to MSDS for additional safety information for specific chemicals.

4 Equipment, Materials and Reagents

- Test tubes, pipettes, or other appropriate containers/items
- Resorcinol
- Concentrated Sulfuric Acid

Resorcinol Reagent, 0.1% (w/v)
Dissolve 0.1 g Resorcinol in 100 mL water.

5 Standards, Controls and Calibration

Freshly prepared reagent will be quality tested with Sucrose and the results recorded in a retrievable logbook. Performance of reagents will be verified monthly and the results of the checks placed in a logbook. 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. The expected result is an orange to red color at the interface.

6 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 and Supporting Documentation

Cheronis, N. D., J. B. Entrikin, and E. M. Hodnett. 1965. Semimicro Qualitative Organic Analysis. John Wiley and sons, Inc. publisher, p. 390.

Preparer

Raymond A. Waller, Jr. Date: 10/04/2004
Controlled Substance Advisory Board Chair

Concurrence

Forrest Davis Date: 10/04/2004
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	01/01/2005	Original Issue

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 Documents

CS-09-01 Resorcinol Test

CS-07-02 Fourier Transform Infrared Spectrophotometry

3 Safety

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.

Use a glass Pasteur pipette to add the H_2SO_4: **do not** use a mechanical pipettor with concentrated acids.

Refer to MSDS for additional safety information for specific chemicals.

4 Equipment, Materials and Reagents

- Test tubes, pipettes, or other appropriate containers/items
- 1-naphthol
- ethanol
- Concentrated Sulfuric Acid

Modified Molisch Reagent 15% (w/v)
Dissolve 1.5 g 1-napththol in 10 mL ethanol. Store the reagent, protected from light, at room temperature.

5 Standards, Controls and Calibration

Freshly prepared reagent will be quality tested with Sucrose and the results recorded in a logbook. Performance of reagents will be verified monthly and the results of the checks placed in a logbook. 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. The expected result is an orange to red color at the interface.

6 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 and Supporting Documentation

Winton, A. L. , and K. B. Winton. 1945. The Analysis of Foods. John Wiley and sons, Inc. publisher, p.157.

Cheronis, N. D., J. B. Entrikin, and E. M. Hodnett. 1965. Semimicro Qualitative Organic Analysis. John Wiley and sons, Inc. publisher, p. 390.

Menke, J. L. 1974. “Detection of Sugar”. In General Information Bulletin 74-8, National Bomb Data Center, p. 13.

Preparer

Raymond A. Waller, Jr. Date: 10/04/2004
Controlled Substance Advisory Board Chair

Concurrence

Forrest Davis Date: 10/04/2004
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	01/01/2005	Original Issue

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 Documents

Controlled Substances Worksheet (LAB-CS-01)

Acid Analysis Flow Chart (CS-09-03A)

Iodine or Hydriodic Solutions (CS-09-04)

3 Safety

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.

4 Equipment/ Materials/Reagent

- Spot plates, pipettes, or other appropriate containers/items
- pH indicating strips (range of 0-14)
- ~1% Barium chloride ($BaCl_2$) Reagent
- ~5% Silver nitrate ($AgNO_3$) Reagent
- Conc Nitric acid
- Conc 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*.

A. Silver Nitrate ($AgNO_3$) Test

1. Silver nitrate reagent preparation:

   a) Dissolve 0.5 g silver nitrate in 10 mL H_2O.

   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 following conditions exists, proceed to the appropriate test:

   a) If a white precipitate forms, proceed to HNO_3 Solubility Test.

   b) If a yellow precipitate forms, proceed to NH_4OH 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 (HNO_3) Solubility Test (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 NH_4OH Solubility Test.

C. Ammonium Hydroxide (NH_4OH) Solubility Test (sample which formed a white 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 white precipitate.

3. Add several drops of concentrated NH_4OH to the white precipitate.

4. Record any observations.

D. Ammonium Hydroxide (NH_4OH) Solubility Test (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 NH_4OH to the yellow precipitate.
3. Record any observations.

E. Barium Chloride ($BaCl_2$) Test

1. Barium chloride reagent preparation:
   a) *Dissolve 0.1 g barium chloride in 10 mL H_2O.*
   b) *Keep tightly capped and store at room temperature.*
   c) *Quality test reagent with aqueous Na_2SO_4; 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 HNO_3
   a) *White precipitate dissolves - indicates **carbonate ion***
   b) *White precipitate does not dissolve - indicates possible **chloride ion***
3. Solubility in concentrated NH_4OH
   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 or Hydriodic Solutions (CS-09-04)*

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 HNO_3

   c) White precipitate dissolves in concentrated NH_4OH

   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 pH >5 in conjunction with presumptive anion identification may be used to presumptively identify neutralized solutions or neutral salts in solution.
- The procedures may give incorrect or misleading results if more than one anion is present in solution.

8 Literature and Supporting Documentation

Whipple, O. K. Chemical properties and identification of ions.

CRC Handbook of Chemistry and Physics

DPS Internal Validation

Preparer

Larry Todsen Date: 04/24/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 04/24/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	10/10/2006	Original Issue

IODINE OR HYDRIODIC ACID 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 Documents

Controlled Substances Worksheet (LAB-CS-01)

Acid Solutions (CS-09-03)

Acid Analysis Flow Chart (CS-09-03A)

3 Safety

Iodine is a toxic substance that sublimes at room temperature.

4 Reagent

- 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 (CS-09-03) with the solution.

5.2 Liquid Sample

A. Determine the pH, miscibility, and anion identification from Acidic Solutions (CS-09-03).

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. Silver/grey metallic flake or shot.

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 (CS-09-03) where the formation of a yellow precipitate in the Silver Nitrate Test which is not soluble in NH_4OH indicates presence of **iodide ion**

7 Literature and Supporting Documentation

Whipple, O. K. Chemical properties and identification of ions.

CRC Handbook of Chemistry and Physics

DPS Internal Validation

Preparer

Larry Todsen Date: 06/12/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 06/12/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	10/10/2006	Original Issue

RED PHOSPHORUS

1 Scope

Red phosphorus may be used in clandestine manufacturing of methamphetamine via reduction of ephedrine or pseudoephedrine utilizing hydriodic acid or iodine.

2 Related Documents

Controlled Substances Worksheet (LAB-CS-01)

Acidic Solutions (CS-09-03)

Iodine or Hydriodic Solutions (CS-09-04)

Ammonium Molybdate Microcrystal Test (CS-05-05)

3 Safety

Red phosphorus is a slightly toxic, flammable powder that may cause burns upon skin contact.

4 Equipment, Materials, and Reagents

- 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).

D. The ammonium molybdate microcrystal test may also be performed (CS-05-05).

7 Literature and Supporting Documentation

Chappell, J. et al. 1995. Analyses of inorganic components found in clandestine drug laboratory evidence. J. Clandestine Laboratory Investigating Chemists Assoc., 5:19.

Preparer

Larry Todsen Date: 06/12/2006
Controlled Substance Advisory Board Chair

Concurrence

Forrest W. Davis Date: 06/12/2006
Quality Assurance

Version #	Effective Date	Brief Description of Change(s)
00	10/10/2006	Original Issue

TEXAS DEPARTMENT OF PUBLIC SAFETY
CRIME LABORATORY

Controlled Substances Worksheet

LAB-CS-01 Rev.02 (07/2006)

Date Started _____ Lab Case # _____

Date Completed _____ Analyst _____

Gross Inventory Wt _____ Page _____

Outside Container	Inside Container

Exhibit #	# Items	# Analyzed	# Exams	Balance ID	Weights				Marquis	SNP	$Co(SCN)_2$	Microscopic	Duquenois	TLC	FTIR	GC / MS	UV		Other Tests	Class / Substance Identified
					Before Analysis	Tare	Net	After Analysis									Acid	Base		

Additional Notes

TEXAS DEPARTMENT OF PUBLIC SAFETY
CRIME LABORATORY

Marihuana Worksheet

LAB-CS-02 Rev.00 (07/2006)

Date Started _____ Lab Case # _____

Date Completed _____ Analyst _____

Gross Inventory Wt _____ Page _____

Outside Container | Inside Container

Exhibit #	# Items	# Analyzed	# Exams	Balance ID	Weights				Macro/Micro						Duquenois	TLC	Other Tests	Class / Substance Identified
					Before Analysis	Tare	Net	After Analysis	Cystolithic Hairs	Glandular Hairs	Conical Trichomes	Seeds	Stems	Other Features				

Additional Notes

TEXAS DEPARTMENT OF PUBLIC SAFETY
CRIME LABORATORY

Acid Analysis Flowchart

LAB-CS-03 Rev.00 (10/2006)

Lab Case # _____

Initials _____

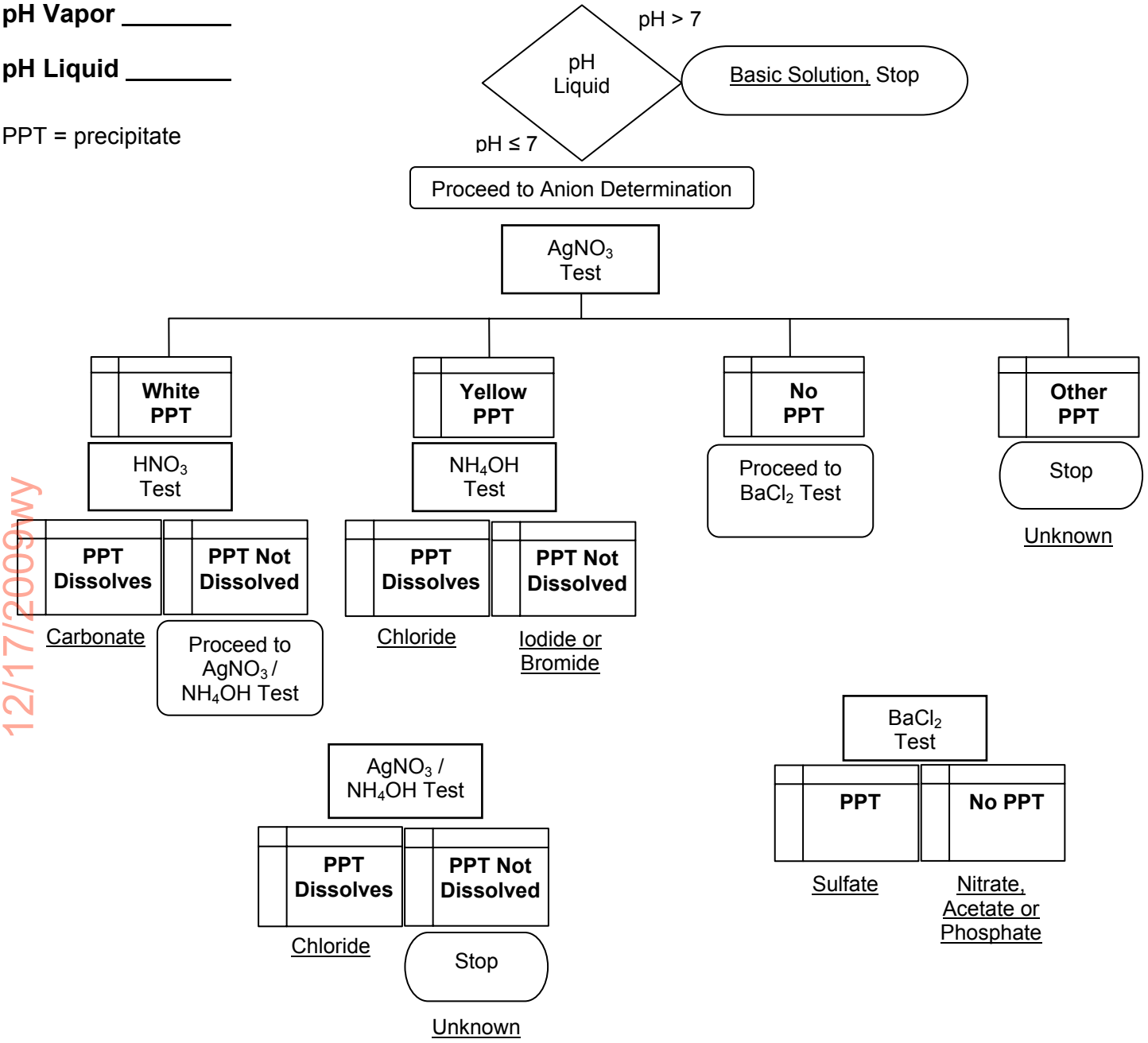
Date _____

Page _____

pH Vapor _____

pH Liquid _____

PPT = precipitate



Preliminary Results of Anion Determination _____