



**Seized Drugs**  
**Standard Operating Procedures**  
Comparative and Analytical Division



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## 1. Goals and Objectives

### 1.1. Goals

1.1.1. The primary goal of the Seized Drugs section (previously referred to as Controlled Substances) is to support the mission of the Houston Forensic Science Center (HFSC) by providing quality analysis of evidence received for the presence of controlled substances including pharmaceutical and illicit drugs, botanical material, related chemicals and paraphernalia as well as dangerous drugs as efficiently as possible utilizing available resources.

### 1.2. Objectives

1.2.1. To maximize efficiency, requests for analysis will be reviewed and the priority status identified (in jail defendants, grand jury or court requests, priority investigations, etc.). The requestor may be contacted at any point prior to or during the analysis to clarify the nature and expected time-line for results of analysis to be completed. Requests that are accepted for analysis will be handled based upon the following objectives:

1.2.1.1. The average turn-around time for requests to be completed should be less than 30 days from the time that the request is received until the report is issued.

1.2.1.2. Investigative priority requests that are associated with on-hold defendants will be assigned to an analyst as soon as possible after receipt of the request with the goal of reporting the results before the end of the day. If analysis including technical review cannot be completed by the end of the day, an attempt will be made to notify the requestor using available information.

1.2.1.3. Other investigative priority requests (search warrant, to be warrant, controlled delivery) will be assigned based upon information and expectations received from the requestor.

1.2.1.4. Evidence associated with requests for defendants who are listed as in jail will be prioritized before requests for defendants who are listed as on bond.

1.2.1.5. Botanical cases (live plants) should be dried as soon as possible once received by an analyst and analyzed within one week once dried.

1.2.1.6. Excess quantity requests should be analyzed within two weeks of assignment to an analyst.

1.2.1.7. All reports should be generated as soon as possible after the completion of the analysis of evidence associated with a request but preferably within two working days.





- 1.2.1.8. All case files should be technically and administratively reviewed within five working days following the generation of the report.
- 1.2.1.9. All evidence should be prepared for return to the submitting agency within five working days of the issuance of the final report.



## 2. Evidence Handling

### 2.1 Scope

2.1.1. To provide guidelines for the handling of evidence in the Seized Drugs section.

### 2.2. Receiving and Documenting Evidence

2.2.1. It is the responsibility of the analyst to maintain the integrity of the evidence at all times while in his/her custody. All evidence must be protected from loss, cross-transfer, contamination and/or deleterious change.

2.2.2. All evidence received by an analyst is to be assigned by the section manager, section supervisors, or designee and must be documented as follows:

2.2.2.1. The analyst will examine the evidence container(s) to ensure that proper seal(s) are in place. A proper seal is one in which there is no possibility that the contents of a container can be removed, altered or a substitution made without the seal being obviously disturbed.

2.2.2.2. Receipt of evidence will be documented at the time of transfer either electronically or on paper as part of the chain of custody.

2.2.2.3. Each outer container (bag, envelope, box, etc.) must be marked with a unique case identifier and the analyst's initials. The outer container is usually an evidence envelope, but it can be anything that contains exhibits for a case. The unique case identifier may be a historic lab number, an agency case number, or a forensic case number depending upon when the case evidence was received or analysis was requested. In addition, an item designator is used with the unique case identifier to distinguish items within a case.

2.2.2.4. A submission form is usually filled out for all seized drug evidence submitted. If a submission form is not available, pertinent information such as the agency case number, suspect name(s), or a description of the evidence submitted may be obtained from available sources or by contacting the officer directly.

2.2.2.5. The contents of items of evidence that are opened will be inventoried and compared with the documentation on the submission form (or equivalent). The analyst will itemize the actual evidence received on the **Inventory Sheet** and include the following information:

2.2.2.5.1. The unique case identifier, the start date for the inventory of the listed parent item(s), analyst initials, and page number.



- 2.2.2.5.2. A description of the exhibits within parent items along with the corresponding sub-item numbers. The descriptors can include color, material type, package type, size, and count. Sub-items will be grouped based on the appearance of the packaging, contents, and analytical scheme (see Analysis Guidelines section).
- 2.2.2.5.3. The use of abbreviations is acceptable as long as they are commonly used or are included in the Abbreviations section.
- 2.2.2.5.4. For large numbers of sub-items it is acceptable to describe them as numerous instead of determining an actual count.
- 2.2.2.6. Items of evidence that are received but not opened (and therefore not inventoried) will be noted as such on the **Inventory Sheet** and will be documented on the final report along with inventoried evidence.
- 2.2.2.7. All exhibits contained within an inventoried parent item will be labeled with the analyst's initials and the unique case identifier and item designators. In a case with numerous small items grouped together, such as small ziplocks, the exhibits may be placed in a container such as a ziplock on which the analyst has placed the unique case identifier and item designators and his/her initials. If during testing a difference is noted, then the small items will be grouped appropriately and analyzed and labeled separately.
- 2.2.2.8. Sometimes it is necessary to recopy inventory notes or to re-itemize evidence. This may happen when analytical results show that a sub-item needs to be further divided because the exhibits are not homogeneous. It may also happen when a request for testing of unopened (not inventoried) items is received. In situations such as these, the original inventory documentation will be retained as part of the case record. It is acceptable to strike out the original notations by drawing a single line through them and initialing it.
- 2.2.2.9. If there are significant discrepancies in submission documentation or with evidence received, then a section supervisor is to be notified as soon as possible. Discrepancies may include mismatched suspect names, incorrect agency case numbers, mismatched evidence, or apparent missing evidence. The discrepancy may simply be the result of writing or typing the information incorrectly or the submitting officer may have inadvertently switched items of evidence.
- 2.2.2.10. It is sometimes necessary to contact the submitting agency to determine the cause of a discrepancy. In the case of missing evidence the submitting officer, the submitting agency, and the HFSC Division Director may all need to be contacted. If discrepancies with evidence need to be corrected by the submitting officer, then the evidence condition will be documented by the receiving analyst and verified by a section



supervisor. The evidence will be returned to the submitting agency for correction before analysis proceeds.

2.2.2.11. Discrepancies and attempts to clarify them through available information will be documented as part of the case file and will be included in the report.

### **2.3. Cases Containing Currency, Valuables, Large Items, and Bullets**

2.3.1. All U.S. currency, valuables, large items, and bullets will be prepared by the analyst for transfer back to the submitting agency. Do not write on currency to allow its eventual return to general circulation. Record the serial number(s) or photocopy any paper U.S. currency. According to Federal Regulations, photocopies of U.S. currency are permissible provided that the reproduced items are less than three-quarters or greater than one and one-half times the size of the part being reproduced.

### **2.4. Cases Requiring Examination for Latent Prints**

2.4.1. Generally, latent print requests are made by the submitting officer on the submission form when evidence is submitted for analysis. In addition, Assistant District Attorneys (ADA's) or defense attorneys (through a court order) may request that any or all items in a case be examined for latent prints. If a case has already been analyzed for seized drugs when the print request is made, the analyst will inform the person making the request that the evidence has already been handled so that the requestor can determine if prints are still needed.

2.4.2. In some cases, evidence will be processed for latent prints prior to being received by a seized drug analyst. In other cases, the seized drug analyst will prepare the evidence for transfer to the Latent Print section by separating the packaging from the materials (powder, plant substance, etc.). If desired a Latent Print analyst will be made available to offer guidance and coordinate the separation and collection of item packaging. Analysts should always wear gloves and handle the evidence as little as possible. Evidence complete with packaging may be photographed prior to preparation for transfer to the Latent Print section.

### **2.5. Cases Containing Possible Biohazards**

2.5.1. Cases that contain items that could represent a possible biohazard to the analyst require special handling. While working with possible biohazards, proper precautions should be taken including wearing gloves, lab coat, masks, and safety glasses, and taking extra care not to touch any part of your body, especially your face. If your work area should become contaminated, wash the area thoroughly with dilute bleach. Avoid touching uncontaminated surfaces (such as telephones, doorknobs, etc.) with soiled gloves. If you work in the hood, clean thoroughly with dilute bleach when you are finished. Whenever possible use disposable beakers, pipettes, Kimwipes, etc. and dispose in the biohazard container. Anything that is not disposable and has come in contact with bodily fluids



needs to be washed with a solution of dilute bleach (dilute bleach is prepared by mixing one-part commercial bottled bleach to nine parts water).

2.5.2. Some items that require special handling are the following:

2.5.2.1. **Syringes** - remove any exposed needles with the needle cutters. If the syringe needs to be analyzed, then the analyst should determine if the needle should be removed before the analysis begins or wait until after the analysis is completed. If the syringe is not exposed (capped, received in an appropriate biohazard container), then it is not necessary to remove the needle. The analyst will document removal of a syringe needle on the **Examination Sheet**.

2.5.2.2. **Latex pellets** or anything else removed from the stomach or lower bowel - in the hood wash the pellets with a bleach solution while wearing double gloves. All preliminary weighing and sampling of the pellet contents is done in the hood. When you are finished handling the pellets, place them in a ziplock bag. Clean the hood area with dilute bleach solution.

2.5.2.3. Items contaminated with blood or items identified as removed from a body cavity, the toilet, groin, crotch area, etc. could represent a biohazard and should be handled accordingly.

## **2.6. Return of Evidence to the Submitting Agency**

2.6.1. All items and sub-items within a case will be packaged to protect from loss, cross-transfer, and/or deleterious change. Whenever possible, evidence will be repackaged in the same condition as it was received.

2.6.2. If evidence needs to be repackaged (for example, containers are leaking or to assist with viewing in court) all containers added by an analyst will be labeled to indicate that they were not part of the original submission.

2.6.3. Before evidence is sealed, the contents will be checked to ensure that it is properly labeled with the analyst's initials, the unique case identifier, and item designators.

2.6.4. Outer evidence containers will be sealed, and the seal labeled with the analyst's initials and date before being returned to the submitting agency.

## **2.7. Related Documents**

2.7.1. Inventory Sheet

2.7.2. Examination Sheet



### 3. Analysis Guidelines

#### 3.1. Scope

- 3.1.1. 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, botanical material, and other chemical substances.

#### 3.2. Safety

- 3.2.1. Use caution when handling any unknown substance or chemical.
- 3.2.2. For hazardous materials, or possible hazardous materials, use appropriate personal protective equipment including eye protection, gloves, masks, and lab coat.
- 3.2.3. Use proper lifting techniques and caution when handling heavy items.
- 3.2.4. Use caution and proper technique when using sharp instruments to cut into evidence packaging.

#### 3.3. Procedure

- 3.3.1. **Note: Only one case shall be opened at a time for analysis. If the case cannot be completed, it must be secured before another case may be opened (e.g. If you have a priority case that requires immediate attention). This is to ensure that all cases are protected from loss, cross-transfer, or contamination.**
- 3.3.2. The general guidelines for which items in a case need analysis are as follows:
  - 3.3.2.1. If the charge is Possession of a Controlled Substance (PCS) and/or Delivery of a Controlled Substance (DCS), analyze the highest penalty felony substance for each suspect listed. Lower felonies, misdemeanor substances and/or residues may be retained and not analyzed.
  - 3.3.2.2. If the charge is Tampering and only residues are present, then at least one residue per suspect should be analyzed.
  - 3.3.2.3. If the charge is PCS and only misdemeanor substances are present, then analyze the controlled substances present but retain any dangerous drugs (definition of dangerous drug = prescription drugs not listed in any Schedule or Penalty Group).
  - 3.3.2.4. If the charge is "obtain drugs by fraud, possession of a dangerous drug, delivery of a dangerous drug, practicing dentistry/medicine without a license, fraudulent prescription, etc.," then at least one dangerous drug should be analyzed.



- 3.3.2.5. If the charge is "possession of a dangerous drug" and there are both controlled substances and dangerous drugs present, analyze the controlled substances and retain any dangerous drugs without analysis.
- 3.3.2.6. Any items in a case indicated as being seized due to a delivery transaction should be analyzed.
- 3.3.2.7. If there are multiple suspects for a case, it may be necessary to analyze more items than those outlined above. Check all sources of information.
- 3.3.2.8. In each case, the most significant items should be identified and analyzed based on available information. This includes such things as the specific charges or types of offense, items unique to a single suspect, the examinations requested, the descriptions of evidence submitted, as well as the analyst's visual inspection of the items.
- 3.3.2.9. If an analyst consults with a case associated officer, Assistant District Attorney, or an individual with the Grand Jury and they specify which items are needed for prosecution, then all other items in a case may be retained without analysis. Communications will be documented electronically or within the case file.
- 3.3.2.10. Items which are not analyzed will be reported as such.
- 3.3.2.11. In all cases, request for analysis of unanalyzed items by a principal associated with a case may require further analysis.

#### **3.4. Sampling Guidelines**

- 3.4.1. Sampling evidence is an important step in drug analysis. The analyst must be sure that what is sampled is truly representative of the total population. The analyst must take into consideration the homogeneity (or lack thereof) among packaging (bags, bottles, etc.) and the contents (powder, liquid, plant substance, tablets, etc.). For a case that contains multiple containers, group them based on visual examination of the containers and of the contents. **See sections 3.8 – 3.10 for additional information about sampling of tablets and capsules.**
- 3.4.2. The Seized Drugs section uses sample selection as a primary method of selecting items for analysis. In some circumstances there may be a need for a statistical method of sampling for the analyst to be able to make an inference about the entire population.
- 3.4.3. Often evidence submitted for analysis consists of a single package (bag, vial, balloon, etc.) containing a suspect material. For these items, a small amount of material is removed and subjected to the analytical procedures described in this section. The analytical results are considered to be representative of the entire contents of the package.



- 3.4.4. When multiple containers of a suspected controlled substance are submitted to the section for analysis, the analyst must use discretion and perform analysis on the number of packages that is sufficient for that case. Careful visual inspections and personal experience are essential in determining the proper sampling procedure. This may include analyzing enough packages to meet the requirements of the Texas Health and Safety Code.
- 3.4.5. When all items within a group are sampled and are individually identified, no documentation of the sampling plan is necessary on the report.
- 3.4.6. For groups that contain a large number of items an alternative sampling plan based on the hypergeometric distribution will allow the analyst to analyze a portion of the items and subsequently make statistical inferences about the population. This random sampling procedure is a tool, which may be used by the analyst to demonstrate that a statistically significant percentage (90%) of the items sampled are positive to within a 95% confidence level. The following table prescribes the minimum number of items randomly selected from a population to be tested.

Total Number of Items in a group (Population)	Required Number of Consecutive Positives
≤ 10	All
11-13	10
14	11
15-16	12
17	13
18	14
19	15
20-26	16
27	17
28-29	18
30-37	19
38-39	20
40-48	21
49-58	22
59-69	23
70-88	24
89-109	25
110-159	26
160-279	27





280-939	28
940+	29

To use statistical sampling to make conclusions regarding a population the analyst should perform the following steps:

- 3.4.6.1. Determine the total number of items in the population (grouping) to be sampled and record the total net weight of the population on the **Examination Sheet**.
- 3.4.6.2. Use the above table to determine the number of randomly selected items for testing. A true random sample is one selected without bias. Since use of random number tables or computer-generated random numbers may not be practical, an alternate sampling technique such as a “black box” method may be used. This type of method prevents the sampler from consciously selecting a specific item from the population (for example, all units are placed in a container and the items for testing are selected without bias). Record the sampling method and the total net weight of the randomly selected items on the **Examination Sheet**.
- 3.4.6.3. Each randomly selected item is to be analyzed separately and completely.
- 3.4.6.4. If testing indicates a difference in the randomly selected items, then all items in the population (grouping) will need to be analyzed separately or the population will need to be subdivided into separate groups as appropriate.
- 3.4.6.5. Documentation that statistical sampling was used **including confidence levels and corresponding inferences regarding the population** is to be noted on the report.
- 3.4.7. Occasionally it will be necessary to perform additional testing beyond statutory requirements of the Texas Health and Safety Code. This may be at the request of an officer for investigative purposes or by an ADA for enhancement. Under these circumstances, the following non-statistical sampling plan may be utilized:
  - 3.4.7.1. A sample is taken from each item and identified by individual screening tests and composite GC/MS testing. This type of analysis will be documented on the report.
- 3.4.8. Regardless of the sampling technique used, if one negative sample is found mixed with items containing a controlled substance, or if a different controlled substance or dangerous drug is indicated, then all items must be analyzed separately, or other special sampling techniques must be applied.



### 3.5. Basic Analytical Scheme (Powders, Liquids, Tar and Chunk Substance)

- 3.5.1. The analyst must determine the appropriate sampling techniques, methods of recovery, extraction procedures, and methods of analysis to be used for the identification of a substance on a case-by-case basis.
- 3.5.2. One positive structural elucidation instrumental test (either FTIR or GC/MS) and at least one other different positive test (including chemical spot tests, pharmaceutical identification, TLC, UV/VIS, GC/FID, GC/MS or FTIR) is required for identification of an unknown substance. The combination of tests chosen must identify the specific substance present and must eliminate the possibility of a false positive identification.
- 3.5.3. When possible, separate sample portions should be used for testing. For example, one portion from an exhibit would be used for chemical spot tests and a separate portion would be used for GC/MS. In some cases, it may not be practical to use separate portions such as when there is limited sample, and this should be documented in the case notes. For example, when TLC is performed on the same portion used for GC/MS this is noted on the **TLC Sheet**.
- 3.5.4. Data required for instrumental analyses
- 3.5.4.1. Maintenance and quality assurance procedures are documented and are available for each instrument within the section. It is the analyst's responsibility to verify that an instrument is working properly before use.
- 3.5.4.2. The data generated from an instrumental method must be documented with the unique case identifier and item designators and the analyst's handwritten initials on every page. The date on the printouts will serve as the date of observation unless otherwise noted by the analyst. The following will also be documented:
- 3.5.4.2.1. UV  
All appropriate information regarding sample preparation, wavelengths, and absorbances will be documented on the UV printout or in the case file.
- 3.5.4.2.2. GC/MS  
All appropriate information regarding sample preparation, retention times and library/literature comparisons will be documented on the GC/MS printouts or in the notes. Blanks run prior to the samples will be maintained with the case file.
- 3.5.4.2.3. FTIR  
All appropriate information regarding sample preparation and library/literature comparisons will be documented on the FTIR printouts or in the case file.



#### 3.5.4.2.4. GC/FID

All appropriate information regarding sample preparation, retention times, weights, or calculations will be documented on the GC/FID printouts or in the case file.

3.5.5. Non-instrumental methods may be used to aid in the analysis of powders, liquids, tar, and chunk substance. These methods may include the following tests:

##### 3.5.5.1. Thin Layer Chromatography

The conditions, standards used for comparison, and results for all TLC runs will be documented on the **TLC Sheet**. The final results will also be noted on the **Examination Sheet**.

##### 3.5.5.2. Chemical Spot Tests

Any reaction observed by the analyst is documented on the **Examination Sheet** by writing the color observed. In addition, the performance of blank controls and spot plate checks are documented on the **Examination Sheet**.

3.5.6. A total net weight is determined and recorded for all powders, liquids, tar, and chunk substance to be reported. An exception is for liquids in an abusable volatile chemical case where a weight does not need to be determined. If the net weight is at a cut-off for a penalty threshold, then sufficient significant figures will be recorded and reported to ensure that the correct weight range is determined. The balance used to determine the weight will be indicated on the **Examination Sheet**.

3.5.7. It is common for abusable volatile chemicals (such as toluene) and phencyclidine (PCP) liquids having an ether-based solvent to evaporate rapidly so these cases should be analyzed on a priority basis. Because of this evaporation, a weight obtained by the analyst may be less than the listed weight as submitted.

### 3.6. Drug Residues

3.6.1. Residues are samples which are either too small to be weighed accurately or that which remains after the bulk has been removed. Residues can be sampled by mechanical means (e.g. shaking or scraping) or chemical means (e.g. rinsing with solvent).

3.6.2. A small amount of the residue is removed for analysis, ensuring that enough residue remains for an independent analysis. A good rule of thumb is to use less than half of the total sample.

3.6.3. If visual examination of evidence which is needed for charges indicates that the amount of sample/residue is too small to retain a sufficient sample for reanalysis, then the item will be examined by another analyst to confirm the lack of available sample. Both analysts



will initial the observations on the **Examination Sheet**. The item is to be reported as “No analysis performed (Insufficient sample for analysis and retesting)”.

3.6.4. If visual examination of evidence which is needed for charges indicates that no sample/residue is present for analysis, then the item will be examined by another analyst to confirm the absence of sample. Both analysts will initial the observation on the **Examination Sheet**. The item is to be reported as “No analysis performed (no visible sample)”.

3.6.5. When field testers are received without any other evidence to analyze, they will be reported as “No unprocessed sample available for analysis.”

3.6.6. If a request is received to analyze evidence that has been or would otherwise be reported as “insufficient sample”, “no visible sample”, or “no unprocessed sample”, then the requestor’s name, contact information, and position will be documented. The section manager, a section supervisor, or designee will be contacted to provide directions on how to proceed.

If it is determined that analysis will be conducted on these items, then procedure blanks will be performed for the tests conducted. Procedure blanks verify that glassware, solvents, reagents, and instruments are clean prior to the analysis of these samples. Documentation of procedure blanks will be included in the case notes.

Any procedure blank vials and/or sample extract vials that remain following analysis will be evaporated to dryness, labeled appropriately, and retained with the case evidence.

3.6.7. Analysis of residues will follow the basic analytical scheme noted in section 3.5.

3.6.8. The weight for residue samples will be noted as “trace” on the **Examination Sheet**.

### 3.7. Plant Substance and Plant Substance Residues

With the passage of HB 1325 (effective June 10, 2019) the definition of marijuana per Texas Health and Safety Code Section 481.002 (26) means the plant Cannabis sativa L. with certain exclusions including hemp as that term is defined by the Texas Agriculture Code Section 121.001. **These definitions require that for a plant sample to be identified as marijuana it must be shown to contain delta-9-tetrahydrocannabinol (THC) at a concentration of more than 0.3%.**

3.7.1. Plant substance samples received for testing may be identified as Cannabis sativa L. **or marijuana** but may also be plant material that has been combined with other substances such as synthetic cannabinoids, PCP, or cocaine. The analyst may have to use a combination of **instrumental and non-instrumental** techniques **to determine if plant**



substance samples are or if they contain a controlled substance (see the basic analytical scheme noted in section 3.5).

- 3.7.2. For the identification of *Cannabis sativa* L., positive microscopic identification, identification of at least one cannabinoid by GC/MS decision-point assay, and at least one other different positive test (including Duquenois-Levine chemical spot test or TLC) are required.

Any features observed during microscopic examination of samples will be documented on the ***Cannabis sativa* L. Checklist**. For a microscopic examination to be positive a minimum of 2 physical characteristics must be observed including cystolithic hairs or glandular hairs. Generally, sample portions used for microscopic examination are also used for additional testing and this practice is not documented in the case notes.

For *Cannabis sativa* L. to be further identified as marijuana a GC/MS decision-point assay must show that delta-9-THC is present and that the concentration is at or above the administrative threshold of 1% (refer to Section 7 for additional details regarding the GC/MS decision-point assay).

- 3.7.3. For mushrooms or plant material suspected of containing psilocin / psilocybin the Weber chemical spot test may be performed to test for the presence of psilocin / psilocybin. If the Weber test is positive, then a positive structural elucidation instrumental test (GC/MS or FTIR) must be performed to report the presence of psilocin / psilocybin.

- 3.7.4. Live plants:

3.7.4.1. Fresh plants are dried before weighing and analyzing to prevent decomposition.

3.7.4.2. Remove roots, dirt and mature stalks before weighing. Mature stalks are the main axis of the plant, fluted in appearance, and are greater than ~1 centimeter in diameter or larger. Stems are also fluted in appearance and serve as a support structure for another part of the plant such as a leaf or flower and do not have to be removed.

3.7.4.3. The weight for the dried plants will be significantly less than the listed weight as submitted.

- 3.7.5. Handling of seeds

Generally, seeds that are received in the absence of additional material are retained without analysis as to their type but may be analyzed to ensure that they do not contain



or have not been combined with a controlled substance. Seeds that are mixed with other material may be left as part of that material for purposes of weighing and analysis.

3.7.6. A weight is determined and recorded on all plant substance items that will be reported including cigars, cigarettes, cigar stubs, and cigarette stubs. The weights determined for cigars and cigarettes should not include the weight of the wrapper (paper or tobacco leaf). At least one cigar or cigarette should be opened completely to determine the appropriate wrapper weight to subtract from the total sample weight. If cigar stubs and cigarette stubs need to be analyzed, the weight of the paper may be included in the total weight and this is to be indicated both on the report and on the **Examination Sheet**. If the weight of the cigarette stubs or cigar stubs makes a difference to the weight cut-offs as listed in the *Texas Controlled Substances Act*, then the paper should be removed. Pipes and residues are not weighed. If *Cannabis sativa L.* or **marihuana** weights are determined in metric units, they will be converted to ounces or pounds for the report.

3.7.7. For the analysis of suspected *Cannabis sativa L.* or **marihuana** the weight of plant substance must be at least 0.20 grams. This helps to ensure there is sufficient sample available for reanalysis. If the weight of plant substance is less than 0.20 grams (or trace for a residue), then the item is to be reported as "No analysis performed (Insufficient sample for analysis and retesting)".

3.7.8. In cases where plant substance is contaminated with an identified controlled substance such as cocaine, phencyclidine, or codeine which cannot be easily separated from the plant substance, the total weight is recorded in grams. For cigarettes or cigars dipped in codeine syrup or phencyclidine liquid the entire weight is recorded (including wrapper / paper / and the filter for manufactured items since it is contaminated with the controlled substance).

3.7.9. In cases where plant substance has undergone excessive decomposition, the item should be examined by another analyst and both analysts will initial the observation on the **Examination Sheet**. It is recommended that the evidence be photographed to document its condition. The item is to be reported as "No analysis performed due to excessive decomposition".

### **3.8. Tablets and Capsules – General**

3.8.1. Tablets and capsules are generally identified as pharmaceutical or clandestine products. Pharmaceutical products are those manufactured by legitimate pharmaceutical companies who mark their products with logos which identify both the manufacturer and composition. Clandestine products by contrast are manufactured illegally and may have markings which simulate legitimate products, but usually they are distinctive logos that represent commercial products, sports teams, or cartoon characters.



- 3.8.2. Tablets and capsules can typically be grouped based upon their appearance (size, color, and markings) **and/or packaging**. Once separated into these groupings, each tablet and capsule should be considered an individual item for the purposes of sampling.
- 3.8.3. A net weight and number will be determined and recorded for all tablets or capsules that will be reported. If the total number of tablets or capsules in one grouping is too large to count (approximately 20), then it is acceptable to describe them as numerous.
- 3.8.3.1. If a statistically based sampling plan is used, then the number of tablets or capsules will need to be established for use as the population from which a random number of samples are taken (see section 3.4.6). It is acceptable to use a weight conversion to approximate the number and include this in the case file documentation. The tablets or capsules may still be described as numerous on the report.
- 3.8.4. For tablets and capsules that require analysis, follow the analytical schemes below based upon whether they can be identified as a pharmaceutical product or not. The combination of tests chosen must identify the specific drug present and must eliminate the possibility of a false positive identification.

### **3.9. Pharmaceutical Tablets and Capsules**

- 3.9.1. The first step in attempting to identify tablets and/or capsules is to compare their markings (logo) with reference sources. If they are successfully identified as pharmaceutical products, this is considered to be an acceptable screening test.
- 3.9.2. Partial tablets may be combined with whole tablets for the purpose of grouping and testing when received packaged together and the characteristics such as markings, color, and shape are consistent with the whole tablets.
- 3.9.3. When performing a pharmaceutical identification, a hardcopy (e.g. computer printout or xerox copy) documenting the source of the comparison will be included in the case file. Pharmaceutical information from packaging (such as blister packs) or manufacturer's information may be used as an acceptable reference source for comparison. The markings (logos) observed by the analyst will be noted on the **Examination Sheet** for comparison.
- 3.9.4. Some pharmaceutical products may not be identifiable by their logos as in the case of new products for which published references are not available. In this case follow the analytical scheme for Clandestine Tablets and Capsules.
- 3.9.5. While partial logos can give useful information as to the possible identity of a pharmaceutical product, they cannot be used as a test for identification. Noting the results of partial logo searches on the **Examination Sheet** is acceptable as long as this is not used as a test. In this case follow the analytical scheme for Clandestine Tablets and Capsules.





3.9.6. When pharmaceutical identification is successful, only one tablet or capsule from each grouping needs to be fully analyzed by performing a structural elucidation instrumental test (GC/MS or FTIR). However, in certain instances such as low dosage products, composite sampling may be necessary for identification. The net weight of the tablet(s) or capsule(s) used will be noted on the **Examination Sheet**, and this type of analysis will be documented on the report.

3.9.7. If any analytical testing procedures indicate that tablets or capsules may be illicit, then pharmaceutical identification is no longer an acceptable test and the analytical scheme for Clandestine Tablets and Capsules will be followed.

### **3.10. Clandestine Tablets and Capsules**

3.10.1. As a result of their clandestine origin, the actual composition of these tablets and capsules can vary greatly from item to item and appearance is generally useful only in grouping the items and is not an acceptable test for identification.

3.10.2. For clandestinely manufactured tablets or capsules, the following options are acceptable for sampling:

3.10.2.1. All tablets (capsules) within a group are sampled and are individually identified using the basic analytical scheme noted under section 3.5. No documentation of the sampling plan is necessary on the report.

3.10.2.2. Use of statistical sampling based on the hypergeometric distribution as noted under section 3.4.6.

3.10.2.3. For each grouping of tablets (capsules) to be reported, each item up to 29 is sampled for individual screening and a composite taken for GC/MS. For groupings with 30 or more tablets (capsules) it is at the analyst's discretion as to whether or not to sample more than 29 items for individual screening and a composite GC/MS. The net weight and number of the tablets (capsules) sampled will be noted on the **Examination Sheet**, and this type of analysis will be documented on the report.

3.10.3. If the analyst has any questions regarding the sampling or analysis of clandestine tablets (capsules) he/she should consult with the section manager, section supervisors or designee.

### **3.11. No Controlled Substance Identification**

3.11.1. Before an item can be reported as "No Controlled Substance Identified", a GC/MS sample will be run.





- 3.11.2. If the presence of a controlled substance is identified in a sample by GC/MS, but a second different positive test cannot be obtained, then the item may be reported as "No Controlled Substance Identified". This may be the result of insufficient sample or the presence of compounds which interfere with additional testing.
- 3.11.3. If an initial GC/MS sample run is negative (no measurable peaks in the Total Ion Chromatogram), then a second more concentrated sample will be run. This can be achieved either by the use of additional sample or by evaporation of the initial sample. The analyst will document sample preparation steps in the case file.
- 3.11.4. If the peaks present in a GC/MS sample run do not indicate the presence of a controlled substance or they are identified as being non-controlled substances (e.g. lidocaine, caffeine), then the item may be reported out as "No Controlled Substance Identified" without an additional GC/MS sample run. However, if a controlled substance peak is indicated but cannot be positively identified, then a second more concentrated sample should be run as described above.

If an initial GC/MS sample run shows the presence of acetaminophen without a controlled substance, then an additional GC/MS sample run is required. This additional run should be prepared using a new larger sample portion and an appropriate solvent such as a mixture of CH<sub>2</sub>Cl<sub>2</sub>/MeOH, CH<sub>2</sub>Cl<sub>2</sub> with a base extraction, or hexane with a base extraction. It is also recommended that an instrument with low split ratio be used to increase sensitivity. These steps will help ensure that a controlled substance such as codeine, hydrocodone, or oxycodone is not being missed.

- 3.11.5. If the only substance(s) identified by FTIR are non-controlled (e.g. lidocaine, caffeine) or cannot be identified, then GC/MS testing will be performed before reporting the results to ensure that a controlled substance is not being masked.
- 3.11.6. A weight does not need to be reported for an item(s) that will be reported as "No Controlled Substance Identified." However, a gross weight and/or a net weight is to be noted in the case file to assist with comparison of items tested and items submitted.

### **3.12. Literature and Supporting Documentation**

- 3.12.1. R.S. Frank, et. al. "Representative Sampling of Drug Seizures in Multiple Containers," *Journal of Forensic Sciences* 36 (1991) pp. 350-357.
- 3.12.2. SWGDRUG Recommendations, 2nd ed. "Part III A - Methods of Analysis/Sampling Seized Drugs for Qualitative Analysis", February, 2006.
- 3.12.3. "Guidelines on Representative Drug Sampling", ENFSI, 2004. [www.enfsi.org](http://www.enfsi.org)



**3.13. Related Documents**

3.13.1. Examination Sheet

3.13.2. Cannabis sativa L. Checklist

3.13.3. TLC Sheet



## 4. Case Documentation

### 4.1. Scope

- 4.1.1. These policies are established as minimum requirements for case documentation and record keeping required for seized drug cases.

### 4.2. Contents of Case Folder

- 4.2.1. Test report on the results of the analysis which has been technically and administratively reviewed and includes the analyst's name, title, and signature.
- 4.2.2. Submission forms or chain of custody records in printed or electronically retrievable format.
- 4.2.3. Section specific forms with information about the exhibits contained in the evidence, any tests performed with the appropriate observations, the results of any analyses, and any other pertinent information including the unique case identifier and item designators, the date for analytical observations and/or tests, and the analyst's handwritten initials.
- 4.2.4. Analytical Data
  - 4.2.4.1. All charts, spectra, and notes will be maintained with the case file. Any photographs should be taped to or digital photos printed on 8 ½" by 11" paper and labeled with the unique case identifier and item designators, the date the photos were taken, and the analyst's handwritten initials (this information may be included within the photograph in lieu of labeling printed photographs). Photographs may also be maintained electronically as part of the case record.
  - 4.2.4.2. All solvent blanks run prior to any case samples for the GC/FID or GC/MS will be maintained with the case file.
- 4.2.5. Any court orders or Motions for Discovery. Alternatively, these documents may be stored electronically as part of the case record.
- 4.2.6. A record of all pertinent phone calls or communications. Alternatively, conversations or activities related to a case may be documented electronically as part of the case record.
- 4.2.7. All documents within a case folder (file) will be labeled with the unique case identifier.



#### **4.3. Technical Review**

- 4.3.1. All examination records and test reports will be technically reviewed by an individual other than the author of the documents under review. This review will include the following:
- 4.3.1.1. Verify that the weights on the report match the weights on the **Examination Sheet**. Check that the weights from submission documentation are consistent with the reported weights.
  - 4.3.1.2. Verify that all spectra support the conclusion(s).
  - 4.3.1.3. Verify that all spectra contain the appropriate unique case identifier and item designators.
  - 4.3.1.4. Verify that all spectra contain any pertinent documentation and that the spectra are documented on the **Examination Sheet**. Check for the presence of any necessary blanks.
  - 4.3.1.5. All **Examination Sheet(s)** and spectra must have the analyst's handwritten initials.
  - 4.3.1.6. Verify that all observations listed on the **Examination Sheet(s)** are consistent with the conclusion(s).
  - 4.3.1.7. Verify that the number of determined weighing events for the total net weights and the corresponding total expanded uncertainties are noted correctly.
- 4.3.2. The completed technical review is documented in the case record.
- 4.3.3. Technically reviewed results, with written approval by the section manager or section supervisors, may be released to a requestor prior to issuing a report in certain circumstances (for example, priority, rush, or investigation cases). The technical review will be documented in the case record, and the release of results will be documented in the report with a description of what was released.
- 4.3.4. After a report has been issued, verbal results may be released by the analyst, section manager, section supervisors, or technically qualified staff. The verbal release of information must be documented in the case record.

#### **4.4. Administrative Review**

- 4.4.1. All case files will be administratively reviewed by an individual other than the author of the report prior to issuance of the report. It is recommended but not required that the



technical and administrative reviews be conducted by different individuals. An administrative review will include the following:

- 4.4.1.1. Verify that both the unique case identifier and the submitting agency number provided are correct for the case being reviewed.
  - 4.4.1.2. Verify all documented weights. It is very important to verify that the weights on the report match the weights on the **Examination Sheet** since this information is used to charge the suspect. Further information regarding weights from submission documentation should also be checked to ensure that the analyst has not put the wrong designation, such as milligrams instead of grams.
  - 4.4.1.3. Verify all spelling, grammar, the unique case identifier and item designators, and the analyst's name and title. Results from all pages of the **Examination Sheet** should be included in the report.
  - 4.4.1.4. Verify that the correct information is listed for the inventoried evidence.
  - 4.4.1.5. Review the chain of custody records for all items of evidence received.
- 4.4.2. The completed administrative review is documented in the case record.

#### **4.5. Technical and Administrative Review Documentation**

- 4.5.1. All changes made to the technical record including those resulting from case review will be documented as part of the case record.
- 4.5.2. Technical and administrative reviews will be documented on the **Case Review Form** which will be included in the case file. This form will include the following information:
  - 4.5.2.1. The unique case identifier and the assigned analyst's initials (the initials may be noted by the reviewer or by the analyst).
  - 4.5.2.2. The reviewer's initials, the date the review was completed, and a Yes/No indication as to whether changes are needed.
  - 4.5.2.3. Space is provided for changes to be described. If no changes are needed, then this space is left blank.
  - 4.5.2.4. Once any changes have been addressed both the reviewer and the analyst will document their acknowledgement on the form with their initials. If there is a disagreement as to the changes, then the section manager, section supervisors, or designee will be consulted to mediate. In this situation, the mediator will include their initials (and additional notes if necessary) when a resolution has been reached.
  - 4.5.2.5. Space is provided on the form for two reviews as in the case where different individuals conduct the technical and administrative reviews.



4.5.2.6. Space is provided for documenting that the chain of custody was included in the review.

4.5.2.7. Space is specifically provided for documenting when GCMS samples are re-run due to an incorrect sample vial being run or when an incorrect case number is recorded.

4.5.3. To ensure the quality of a final report, any significant issues discovered by a technical reviewer (such as reporting a wrong weight, a wrong drug, reporting results without sufficient tests, etc.) must be reported to the section manager, section supervisors, or designee as soon as possible. Administrative review issues should also be reported if it becomes a pattern.

#### **4.6. Report Modification Records**

4.6.1. It is sometimes necessary to modify a report after it has been issued. This may be necessary to correct an error in the report, to document additional analysis conducted after the issuance of the report, at the request of the DA's office, or for various other reasons.

4.6.2. If it becomes necessary to amend a signed report, then the new report will be clearly identified, will contain a reference to the original report that it is replacing, and will clearly state why an amended report was issued. The original report must be maintained within the case record.

#### **4.7. Page Numbering of Examination Documents**

4.7.1. The total number of pages for examination documents within a case file will be indicated on the **Inventory Sheet** along with the date and initials of the person making the notation. If examination documents are added (for example additional analysis is performed), then this information will need to be updated.

4.7.2. Examination documents will include all **Inventory Sheet(s), Examination Sheet(s), Notes Sheet(s)**, instrument printouts, photographs, and other documents produced and used to reach a conclusion.

#### **4.8. Related Documents**

4.8.1. Examination Sheet

4.8.2. Inventory Sheet

4.8.3. Notes Sheet

4.8.4. Cannabis sativa L. Checklist

4.8.5. Case Review Form



## 5. Seized Drugs Worksheets

### 5.1. Scope

5.1.1. To provide guidelines for documentation of tests and observations on the **Examination Sheet**, the **Notes Sheet**, and the **Cannabis sativa L. Checklist**.

### 5.2. Examination Sheet

#### 5.2.1. Case Information

**Case** – This is the unique case identifier which may be an historic lab number, an agency case number, or a forensic case number depending upon when the case evidence was received, or analysis was requested.

**Date** – This is the start date of analysis.

The date for observations that do not have printed data will be documented appropriately if different than the start date.

The date on printouts will serve as the date of observation unless otherwise noted by the analyst.

**Analyst** – Placement of initials in this box indicates the person(s) who performed or observed all of the analysis documented. If an analyst only performs or observes a portion of the analysis, then his/her initials will be noted next to the results for that test.

**Item Number** – The LIMS generated item/sub-item number for the exhibit(s).

**Description** – A brief description of the material may be entered here as well as the number of discrete items. For example, 5 bags with powder may be noted as “5 powder”, 5 bottles with numerous blue tablets may be noted as “5 num tabs”. This is intended to assist the analyst and reviewer with correlating the documentations noted here with the evidence as described on the **Inventory Sheet** (see the Evidence Handling section). This is not a required field but may be used at the analyst’s discretion.

**Page** – The appropriate page number is noted.

#### 5.2.2. Analytical Documentation

**Microscopic** – Pos indicates that a minimum of two physical characteristics for Cannabis sativa L. (including cystolithic and/or glandular hairs) were observed. Neg indicates that insufficient or



no characteristics for *Cannabis sativa* L. were observed. The characteristics observed as well as the number of samples tested will be documented on the **Cannabis sativa L. Checklist**.

**Chemical Spot Tests** – The observations and number of samples tested are documented next to the appropriate named test. Neg indicates that no color reactions were observed.

**Infrequent QC** – When an infrequent chemical spot test is performed a blank space is provided on the sheet to document its name and the observations for the case samples.

In addition, the results of the required quality control check and the drug standard used are noted in the box above.

**Spot Plate Check** – Spot plates are to be visually examined for cleanliness by the analyst prior to use. A check mark on the sheet next to “Spot Plate Check” indicates that the spot plates used were free of residue or debris.

**Blank Checks** - Blank (or negative) controls for all chemical spot tests are performed at the same time as the sample testing. A check mark next to the tests performed indicates that no reaction was observed and that the blank control passed.

**PHI** - The markings (logos) observed by the analyst from pharmaceutical products will be noted. Information obtained from pharmaceutical identifications (see section 11.3.1) for comparison will be recorded appropriately in this box.

**Visual** – Additional observations or notations may be included in this box. Items which do not appear to have residue present may be noted here as “no visible sample” or “NVS”.

**Notes** - If more space is needed for observations or notations then they can be documented in the Notes box as long as the associated items are clearly designated.

Notations regarding the condition of the evidence when received should be included on the sheet (e.g. moldy, wet, apparent blood) as well as any procedures taken which may alter the appearance or weight of the evidence. Examples include removing needles from syringes, drying wet evidence (include length of time dried **before** weighing), drying of fresh plant material (include length of time dried **before** weighing) as well as removal of stalks, roots, and dirt.

When significant quantities of evidence are consumed during analysis, it is recommended that before and after analysis weights are noted on the sheet. Alternatively, note the amount of





sample used for analysis. The before analysis weight is to be reported in such cases. Examples include dilute codeine liquids, large clandestine tablet cases, and samples that are at a cut-off weight.

**UV** – When a sample spectrum is matched to a standard or known substance (based upon peak shapes and maxima), that match is noted. It is not required to include a copy of reference UV/VIS spectra for commonly encountered substances. It may be helpful, however, to include a printed reference spectrum for less commonly encountered substances.

**No acceptable match (or NAM)** should be noted when the sample produces a measurable absorbance, but the spectra cannot be matched to a standard or known substance. This may be due to a significant wavelength shift from expected peak maxima, or interferences from other absorbing substances which cause extraneous peaks or peak shape distortions.

**Negative** should be noted when the sample produces no measurable absorbance, for example: carisoprodol or wax.

**FTIR** – When a sample spectrum is matched to a standard or known substance (based upon peak shapes and maxima), that match is noted. It is required to include a copy of reference IR spectra (usually computer generated from a library search).

**No acceptable match (or NAM)** should be noted when the sample produces a measurable absorbance, but the spectrum cannot be matched to a standard or known substance. This may be due to interferences from other absorbing substances which cause extraneous peaks or peak shape distortions.

**GC/FID** – Document the results obtained.

**GC/MS** – Any identified substances which are to be included on the final report will be noted in the GC/MS box. This includes non-controlled substances if they are to be noted in a footnote (caffeine, lidocaine, nicotine, etc.) and substances necessary to correctly report other controlled substances (acetaminophen and hydrocodone, promethazine and codeine). It is required to include a copy of reference mass spectra (usually computer generated from a library search).

When none of the peaks on the TIC produce mass spectra which can be identified (all **NAM**) or when substances are identified, but none of them will be included on the final report, **Refer to TIC** should be noted in the GC/MS box.

**Negative** should be noted in the GC/MS box when the TIC for the sample produces no measurable peaks.



**TLC** – When a sample spot matches a standard spot (based on color and location), the match is noted along with the number of samples. When the sample and standard spots do not match or are not acceptable for comparison, **Refer to TLC Sheet** will be noted.

**# Exhibits Sampled / Net Weight** – When a sampling plan is used and not all of the exhibits within the group are sampled, then the actual number of exhibits which are sampled will be noted along with their net weight.

**Gross Weight** – Notations of the gross weight will refer to the substance(s) and the inner most container(s) unless otherwise noted.

**Total Net Weight** – This refers to the total net weight of all substance(s) as designated by the item number. It does not include packaging.

**Weighing Events / Uncertainty** – The number of determined weighing events for the total net weight and the corresponding total expanded uncertainty are noted. These values are required for substances identified as controlled substances that have a penalty group threshold weight range.

**Balance(s) Used** – Indicates which balance(s) were used for any weight determinations.

**Results** - The results of the analysis which are to be reported are noted in this box. If the results are negative, then "NCS" or "NCSI" is written.

5.2.3. When a case is reopened and further analysis is required, the following procedures will be followed when the original **Examination Sheet** is used:

5.2.3.1. The date of any additional testing is documented appropriately.

5.2.3.2. If the additional testing is performed by a different analyst, then his/her initials are documented appropriately.

5.2.3.3. Alternatively, a new **Examination Sheet** may be used following the proper guidelines for notations outlined above.



### 5.3. Notes Sheet

**Case** – This is the unique case identifier which may be a historic lab number, an agency case number, or a forensic case number depending upon when the case evidence was received or analysis was requested.

**Date** – This is the date(s) that notations are made. If notations are made on multiple days, then it is acceptable to put multiple dates or date ranges. Any analytical observations must be noted on the **Examination Sheet**.

**Analyst** – Placement of initials in this box indicates the person(s) who performed or observed what is being documented.

**Page** – The appropriate page number is noted.

**Notes** – This sheet is to be used for notations/calculations that do not fit on the **Examination Sheet**. It may be used to document analytical observations as long as the corresponding items, testing conditions, and results are clearly noted. Weights may be recorded or calculated. This sheet may also be used in the processing of Excess Quantity cases.

### 5.4. Cannabis sativa L. Checklist

**Case** – This is the unique case identifier which may be a historic lab number, an agency case number, or a forensic case number depending upon when the case evidence was received or analysis was requested.

**Date** – This is the date that observations are made. Any observations on a different date than this will be documented accordingly next to the corresponding item number.

**Analyst** – Placement of initials indicates the person(s) who made the observations documented.

**Item** – The LIMS generated item/sub-item number for the exhibit(s).

**Stereoscope Used** – The stereoscope used to observe sample characteristics will be noted in the box below the item number.

**Characteristics** – A check mark and the number of samples is placed in each box for the characteristics that are observed. If there are no characteristics observed for a sample, then this will be noted in the appropriate box.



## **5.5. Related Documents**

5.5.1. Examination Sheet

5.5.2. Inventory Sheet

5.5.3. Notes Sheet

5.5.4. Cannabis sativa L. Checklist

5.5.5. TLC Sheet



## 6. Instrument Performance and Maintenance

### 6.1. Scope

6.1.1. The following describes quality assurance guidelines for the maintenance, performance, and repair of analytical instrumentation (and equipment).

### 6.2. General Requirements for Analytical Instrumentation

6.2.1. All instruments will be verified before being placed into service and will be periodically maintained in accordance with the manufacturer's recommendations and specifications.

6.2.2. The performance of all instruments will be re-verified if they are moved or if a major repair is performed. It is the analyst's responsibility to ensure that appropriate re-verification has been done before using an instrument on casework samples.

6.2.3. If an instrument fails calibration or a performance verification check, or if a performance problem is detected during casework, the instrument will be removed from service.

6.2.4. No instrument is to be used if it is not in proper working order. If an instrument is taken out of service, then it will be clearly marked. In addition, if repairs are necessary, then the section manager or designee will be notified.

6.2.5. Records of all repairs and maintenance will be maintained in the section.

6.2.6. Refer to the HFSC Quality Manual for the guidelines regarding retention of performance verification records.

### 6.3. UV/VIS Spectrophotometer

6.3.1. Conduct a performance verification check on UV/VIS instrument quarterly or as needed.

6.3.1.1. Check the wavelength accuracy using the two characteristic wavelength peaks of deuterium light at 486.0 nm and 656.1 nm. Follow the manufacturer's specifications for performing this check. The peak wavelength ranges should be between 485.5 nm - 486.5 nm and 655.6 nm - 656.6 nm respectively.

6.3.1.2. Determine if the instrument meets specifications. If it does not, then the instrument will be taken out of service until the issue can be resolved.

6.3.1.3. Records documenting the results of all performance verification checks will be maintained in the section.



6.3.2. Perform regular and preventive maintenance according to the manufacturer's recommendations. Records documenting all maintenance will be maintained in the section.

#### **6.4. FTIR Spectrometer**

6.4.1. Conduct a performance verification check on the FTIR quarterly or more often as needed.

6.4.1.1. One method is to use the OMNIC ValPro software to check the performance of the instrument. The measurements are made by ValPro utilizing a NG11 Glass Serialized Linearity standard and a 1.5 mil Serialized Polystyrene standard. ValPro tests the spectrophotometer's single-beam energy ratio, noise level, wavenumber accuracy, optical resolution, repeatability and detector linearity. A qualification report is provided to demonstrate the pass-fail results for each test.

6.4.1.2. Determine if the instrument meets specifications. If it does not, then the instrument will be taken out of service until the issue can be resolved.

6.4.1.3. Records documenting the results of all performance verification checks will be maintained in the section.

6.4.2. Perform regular and preventive maintenance according to the manufacturer's recommendations. Records documenting all maintenance will be maintained in the section.

#### **6.5. Gas Chromatography/Flame Ionization Detector (GC/FID) (Rescinded July 2018)**

#### **6.6. Gas Chromatography/Mass Spectrometry (GC/MS)**

6.6.1. The Mass Selective Detector (MSD) will be tuned weekly when in use or more often as needed (e.g. if the instrument is moved or maintenance is performed on the MSD). **The tune will be evaluated** according to established criteria for a successful tune **as noted in the Ideal Standard Tune documents**. It is recommended that **the established criteria** used to check the instrument performance be kept next to the instrument for easy reference.

6.6.2. **Each day that samples are loaded onto an instrument, a** standard check mix will be run and the scan results entered in the instrument logbook and maintained with the tune report for that week. If there is any deviation of the standard m/z ratios, the instrument will be tuned and **a standard mix re-run**.

6.6.3. Printed copies of tune records and **standard** check mix results are maintained in the section.

6.6.4. Run a solvent blank before each sample run and maintain a copy of the blank run with the case file.



6.6.5. Perform regular and preventive maintenance according to the manufacturer's recommendations. Records documenting all maintenance (e.g. column replacement, filament replacement, seal replacement, vacuum oil changes, source cleaning, and major repairs) will be maintained in the section.

## 6.7. Balances

6.7.1. The appropriate balance will be used for the weight being measured. Care should be taken not to overload a balance with too much weight.

6.7.2. Inspect the balances for cleanliness and check the level frequently.

6.7.3. It is the analyst's responsibility to verify that the necessary checks have been performed in the recommended time period for any balances or weights used.

6.7.4. Balances will be calibrated by an external vendor at least annually.

6.7.5. Reference weights will be certified by an external vendor at least annually. Secondary weights will be checked internally at least annually.

6.7.6. Balances will be checked regularly using secondary weights. Balances must be checked whenever they are moved from one location to another.

6.7.6.1. Analytical balances will be checked with secondary weights weekly or as needed.

When the use of an analytical balance is infrequent, performance checks are not required each week if the balance is not used weekly, but a check will be performed prior to each use.

6.7.6.2. Top loading balances will be checked with secondary weights monthly or as needed.

When the use of a top loading balance is infrequent, performance checks are not required each month if the balance is not used monthly, but a check will be performed prior to each use.

6.7.6.3. The bulky balances (high capacity) will be checked with secondary weights prior to use.

6.7.7. To perform regular balance checks the following procedure will be followed:

6.7.7.1. Place the appropriate secondary weight on the balance.

6.7.7.2. Listed below are the acceptable ranges for each balance along with its corresponding check weight(s):



Balance type	Weights	Readability	Acceptable range*
Analytical	100 g	100.0000 g	±0.0005 g
	1 g	1.0000 g	±0.0005 g
Top Loading	2 kg	2000.00 g	±0.06 g
	1 g	1.00 g	±0.06 g
Bulky 4,5	2 kg	2.000 kg	±0.002 kg

\* The acceptable range is determined from the largest expanded uncertainty value (as static weighing is not used to perform regular balance checks) obtained from the historic estimation of the uncertainty of measurement studies.

- 6.7.7.3. If a result from the check is outside of the acceptable range, first ensure that the balance is level and clean and that the weight is centered on the pan prior to rechecking.
- 6.7.7.4. If applicable, use the internal calibration function of the balance prior to rechecking.
- 6.7.7.5. If a result is outside of the acceptable range after performing the actions above, then the balance shall be immediately taken out of service until maintenance and/or calibration are performed by an external vendor.
- 6.7.8. A more extensive internal performance check of the balances must be conducted at least annually, or when a balance is being put back into service, or is being put into service for the first time. The **Balance Performance Check Worksheet** will be used for this purpose.
- 6.7.8.1. The appropriate check weights as listed above are weighed and recorded 10 times.
- 6.7.8.2. The % relative standard deviation (%RSD) is calculated for the recorded weights.
- $$\%RSD = 100 * (\text{standard deviation} / \text{mean})$$
- 6.7.8.3. Each weight reading should fall within the acceptable range as listed on the worksheet. The %RSD must be less than 1%.
- 6.7.8.4. If a result from the check does not meet the acceptance criteria, first ensure that the balance is level and clean and that the weight is centered on the pan prior to rechecking.





6.7.8.5. If applicable, use the internal calibration function of the balance prior to rechecking.

6.7.8.6. If a result does not meet the acceptance criteria after performing the actions above, then the balance shall be immediately taken out of service until maintenance and/or calibration are performed by an external vendor.

6.7.9. Records documenting the results of the balance checks, weight checks, maintenance, and calibrations will be maintained in the section.

### **6.8. Pipettes**

6.8.1. Inspect the pipettes and dispensettes for cleanliness. As needed, clean the inside and outside of them with alcohol wipes.

6.8.2. It is the analyst's responsibility to verify that the necessary checks have been performed in the recommended time period for any pipets or dispensettes used.

6.8.3. Fixed volume and variable volume pipettes and variable volume dispensettes will be calibrated by an external vendor prior to being put into service and at least annually while in service.

6.8.4. If a pipette or dispensette requires maintenance, it will be calibrated by an external vendor before being placed back into service.

6.8.5. Records documenting the results of maintenance and calibrations will be maintained in the section.

### **6.9. Malfunction of an Instrument, Pipette, or Balance**

6.9.1. If an instrument, pipet, or balance fails the performance check or a performance problem is detected during routine maintenance, it must be clearly labeled and removed from service, the section manager or designee must be notified, and the problem recorded.

6.9.2. No instrument, pipet, or balance is to be used if it is not in proper working order.

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

6.9.4. Records documenting repairs and maintenance will be maintained in the section.

### **6.10. Related Documents**

6.10.1. Instrument Logbooks



6.10.2. Balance Performance Check Worksheet

6.10.3. Equipment Service Form

6.10.4. **Ideal Standard Tune for Agilent 3, 4, and 5**

6.10.5. **Ideal Standard Tune for Agilent 6, 7, and 8**



## 7. Gas Chromatography/Mass Spectrometry (GC/MS)

### 7.1. Scope

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

### 7.2. Safety

7.2.1. Use appropriate eye protection, gloves and lab coat when handling solvents, acids/bases, and volatile chemicals. Refer to the SDSs for additional safety information for specific chemicals.

7.2.2. Properly secure high-pressure gas cylinders

7.2.3. Use caution around hot surfaces such as oven interiors and injection and detector ports.

7.2.4. Discard all chemicals and any other pertinent materials in an appropriate manner.

### 7.3. Equipment, Materials, and Reagents

7.3.1. Gas chromatograph/mass spectrometer analytical instrument

7.3.2. Auto-sampler vials and caps

7.3.3. Solvent(s) appropriate for the substance being analyzed as well as acids/bases used for extractions

7.3.4. Derivatizing agents such as N,O-Bis(trimethylsilyl)trifluoroacetamide (BSFTA)

7.3.5. Microliter syringe (where applicable)

### 7.4. Standards, Controls, and Calibration

7.4.1. 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.

7.4.1.1. The instrument will be tuned weekly when in use **or** more frequently as deemed necessary. **The tune will be evaluated according to established criteria for a successful tune as noted in the Ideal Standard Tune documents.**

7.4.1.2. Printed copies of tune records are maintained in the section. **If a successful tune cannot be achieved**, the instrument will be taken out of service until instrument maintenance is performed. Documentation of the problem will be maintained in the section.



7.4.1.3. Although the monitoring of environmental ions such as water, oxygen, and nitrogen are included in the **Ideal Standard Tune** documents, the monitoring of these ions serves as a preventive measure because these ions are a guide to determining if there are leaks and/or water present in the system that could shorten the life of the column and/or the filaments. The presence of these ions does not prevent a tune from being deemed acceptable.

7.4.2. Each day that samples are loaded onto an instrument, a standard check mix will be injected to verify instrument performance. The standard printout will be maintained with the appropriate tune report. If the standard run does not provide acceptable mass spectral identifications, the instrument should be retuned and a standard mix rerun. If the standard still does not provide acceptable mass spectral identifications, then the instrument will be taken out of service until instrument maintenance is performed. Documentation of the problem will be maintained in the section.

If samples are still running from the previous day and there is not an issue with the instrument, then the samples will be allowed to finish running without performing a standard check mix injection. Once the samples have finished running, a standard check mix will be injected and reviewed prior to loading more samples.

If there is an issue with the instrument (and samples from a previous day did not run) or if an issue develops before samples from a previous day have finished running, then the issue will be resolved, and a standard check mix will be injected and reviewed for acceptability. Any samples that did not run can then be reloaded.

7.4.3. Solvent blanks prepared from the same solvent used to prepare samples will be injected between case samples to verify that the solvent, column and syringe are free of contamination. The solvent blank will be run on the same method as the sample and immediately before it.

7.4.4. A procedure blank will be run for samples that will be completely consumed by analysis to verify that the column, acids/bases used for extractions, solvents, and laboratory glassware used are clean prior to the analysis of case samples. A procedure blank for GC/MS analysis will be prepared in exactly the same manner as the sample including the use of the same non-disposable glassware and solvents. The procedure blank is to be run on the GC/MS immediately prior to and using the same method as the sample run. Documentation of procedure blanks will be included in the case notes. If any sample remains after analysis, then the procedure blank vials and sample vials used will be evaporated to dryness, labeled appropriately, and retained with the case evidence.

7.4.5. Any significant peaks in the blank chromatograms will be properly investigated to identify their source (e.g. column breakdown, vial septa bleed, carryover from previous sample run, or instrumental contamination) so that appropriate action (such as replacing solvents



or performing instrument maintenance) can be taken as necessary. Any affected case samples and associated blanks should be rerun (this is not necessary in the case of minor peaks identified as column breakdown or vial septa bleed).

7.4.6. For less frequently encountered controlled substances, standards should be run within the same timeframe that the evidence sample is tested, and a copy of the standard run should be retained in the case file. Examples of less frequently encountered substances include LSD, psilocin, or methaqualone. An acceptable timeframe for running the samples and standards would be within 30 days as long as instrument conditions had not changed (column replacement or method modifications). Available and verified standards are a requirement for this practice.

## **7.5. Procedure**

### **7.5.1. Sample Preparation**

7.5.1.1. Most samples can be dissolved directly into a suitable organic solvent such as dichloromethane, chloroform, or methanol.

7.5.1.2. Some samples will require aqueous extraction into a suitable organic solvent to improve solubility and/or chromatography. The analyst should be careful to remove any aqueous solvents before the samples are injected into the instrument.

7.5.1.3. Some samples will require filtration to remove undissolved solids before they are injected into the instrument.

### **7.5.2. Derivatization**

7.5.2.1. Some substances are not readily analyzed by GC/MS as they are thermally labile and may breakdown in the injector. Other substances may be too polar resulting in broad, ill-defined peaks. Most of these substances may be made more stable or less polar by derivatization. The substances that readily derivatize with silylation reagents such as BSFTA will have an acid, alcohol, or secondary amine functional group.

#### **7.5.2.2. Silyl Derivatization Procedure**

7.5.2.2.1. Dissolve or extract the sample in a suitable aprotic solvent such as acetonitrile (ACN) and place in a GC/MS vial.

7.5.2.2.2. Add the derivatizing agent such as BSFTA to the vial. Typically, a few drops will be sufficient.

7.5.2.2.3. Place the vial in an oven at approximately 65°C for approximately 20 minutes.



7.5.2.2.4. Prepare a derivatization blank following the same procedure as the sample including solvent, derivatizing agent, heating temperature and time.

7.5.2.2.5. Inject the derivatized sample on the GC/MS as usual being sure to run the prepared blank immediately prior to and using the same method as the sample.

7.5.2.2.6. Document the derivatization conditions in the case file.

### 7.5.3. GC/MS Operating Conditions

7.5.3.1. Methods have been developed using appropriate temperature programs and other critical parameters to ensure that the suspected substance(s) will elute during data collection. The methods should allow a reasonable time for unknown or unexpected compounds to elute.

7.5.3.2. Lists of methods with standard retention times and method parameters are available by each GC/MS instrument or are electronically retrievable. The lists provide guidance for the selection of the appropriate method for the compound(s) being analyzed. These lists will be updated annually or more frequently as needed (for example following column changes or method modifications).

### 7.5.4. Analysis and Interpretation

7.5.4.1. The results of the GC/MS analysis for samples and corresponding blank runs will be evaluated, printed, and included in the case file. The printouts will be labeled with the unique case identifier, item designators, and the analyst's handwritten initials. Spectra or notes will also include the date of observation and the method of sample preparation (if not listed on the **Examination Sheet**).

7.5.4.2. The Total Ion Chromatogram (TIC) for each sample will be evaluated first by noting whether the total run time is as expected for the method used. Instances may occur in which the solvent delay was over-ridden, but the resulting data is still acceptable. It may also be observed that the run ended early which could result in the sample being re-run. The TIC will also be evaluated by noting the presence or absence of peaks. The complete absence of peaks may indicate that another solvent or method needs to be used or that there was an error during injection. Peaks present on the TIC will be examined for symmetrical shape, abundance, baseline separation, and possible co-elution. The analyst will determine which peaks to evaluate for mass spectral identification based on the analytical scheme and circumstances of the case and will label these peaks with the identification from the corresponding mass spectra or "NAM" (for No Acceptable Match).



7.5.4.3. Mass spectra will be evaluated for suitability of comparison by noting the presence, abundance, and ratios of ion fragments including base peak, possible molecular ion peak, and extraneous peaks that can result from column bleed or co-elution.

7.5.4.3.1. The analyst will determine mass spectral identifications by comparing the unknown mass spectral fragmentation patterns to those of known standards. The source for the comparison standard mass spectra, typically a stored library or a literature source, will be documented in the case file. 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 analyst verification of mass spectral fragmentation patterns when making an identification.

7.5.4.3.2. Mass spectra for peaks that do not have an acceptable reference comparison (labeled on the TIC as "NAM") will be printed and labeled as "NAM". The printout may be done manually or as part of a method's automatic data analysis.

7.5.4.3.3. Peaks from the TIC that are not evaluated will not have printed mass spectra.

7.5.4.3.4. If a background subtraction is performed for a peak mass spectrum, then a copy of the original mass spectrum is retained in the case file as well as the background subtracted mass spectrum. The retention time used to generate the background subtracted spectrum is noted on the printout.

## **7.6. Limitations**

7.6.1. When analysis by GC/MS is unable to provide positive identification, another technique such as FTIR must be utilized to provide positive identification.

7.6.2. 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 or rearrange before they are ionized, preventing their identification.

7.6.3. It may be difficult to identify individual compounds in a homologous series (straight chain hydrocarbons, fatty acids).

## **7.7. Advantages**

7.7.1. Generally, mass spectra of compounds of interest are specific to single compounds and may be used for positive structural identification.

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



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

7.7.4. A GC/MS auto-sampler increases the efficiency of analysis of numerous samples by functioning unattended.

#### **7.8. Literature and Supporting Documentation**

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

7.8.2. F. W. McLafferty, Interpretation of Mass Spectra, 4th Edition, (Sausalito, California: University Science Books, 1993).

7.8.3. Jehuda Yinon, Forensic Mass Spectrometry, (Boca Raton, Florida: CRC Press, Inc., 1987).

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

7.8.5. R. E. Ardrey, "Mass Spectrometry" in Clarke's Isolation and Identification of Drugs, (London: The Pharmaceutical Press, 1986), 251-263.

#### **7.9. Related Documents**

7.9.1. Instrument Logbook

7.9.2. Examination Sheet

7.9.3. **Ideal Standard Tune for Agilent 3, 4, and 5**

7.9.4. **Ideal Standard Tune for Agilent 6, 7, and 8**





## **8. Gas Chromatography/Mass Spectrometry (GC/MS) Decision-Point Assay for delta-9-Tetrahydrocannabinol (THC) in Plant Substance (New section)**

### **8.1. Scope**

8.1.1. An instrumental analytical technique used for the characterization and structural identification by mass spectral fragmentation patterns of unknown substances including but not limited to natural cannabinoids found in the plant species *Cannabis sativa* L. The assay also uses an administratively determined threshold for delta-9-tetrahydrocannabinol (THC) at 1% as part of an analytical scheme for evaluating whether plant substance samples are marijuana.

### **8.2. Safety**

8.2.1. Use appropriate eye protection, gloves and lab coat when handling solvents, acids/bases, and volatile chemicals. Refer to the SDSs for additional safety information for specific chemicals.

8.2.2. Properly secure high-pressure gas cylinders

8.2.3. Use caution around hot surfaces such as oven interiors and injection and detector ports.

8.2.4. Discard all chemicals and any other pertinent materials in an appropriate manner.

### **8.3. Equipment, Materials, and Reagents**

8.3.1. Gas chromatograph/mass spectrometer analytical instrument

8.3.2. Auto-sampler vials and caps

8.3.3. Culture tubes with caps

8.3.4. Methanol (MeOH) for sample and standard preparation

8.3.5. Pipettes and/or Dispenser

8.3.6. Analytical Balance

8.3.7. Volumetric flasks (Class A)

8.3.8. Delta-9-THC standard or certified reference material (CRM)

8.3.9. Deuterated delta-9-THC standard or certified reference material (CRM) to be used as internal standard



## 8.4. Standards, Controls, and Calibration

### 8.4.1. Preparation of Standard Solutions

#### 8.4.1.1. **0.05 mg/mL delta-9-THC Standard Solution**

Using a volumetric pipette, transfer 500  $\mu$ l of a 1 mg/mL delta-9-THC standard to a 10 mL volumetric flask. Bring to volume with MeOH. Equivalent dilutions should be performed if solutions are prepared on a different scale. **This solution is equivalent to the 1% delta-9-THC in plant extract decision-point.**

#### 8.4.1.2. **0.1 mg/mL delta-9-THC-D3 Internal Standard Solution (ISS)**

Using a volumetric pipette, transfer 1000  $\mu$ l of a 1 mg/mL delta-9-THC-D3 standard to a 10 mL volumetric flask. Bring to volume with MeOH. Alternatively, the ISS may be purchased at the correct concentration and used as supplied. Equivalent dilutions should be performed if solutions are prepared on a different scale.

#### 8.4.1.3. **0.05 mg/mL Secondary Standard Solution**

This solution is prepared from a different delta-9-THC standard (different lot number or vendor) than the solution prepared in 8.4.1.1, but in the same manner. The standard used for this solution may contain additional compounds such as cannabidiol or cannabinol.

8.4.1.4. Standard solutions will be labeled with the name and concentration of the solutions, date of preparation, and the initials of the analyst who prepared them. Standard solutions will be stored in the freezer when not in use.

8.4.1.5. Document the preparation of standard solutions in the case record.

### 8.4.2. Preparation of Controls

#### 8.4.2.1. **Positive Control (Decision-Point Control)**

To prepare the positive (decision-point) control mix equal volumes of the 0.05 mg/mL delta-9-THC Standard Solution and the 0.1 mg/mL ISS.

#### 8.4.2.2. **Secondary Control**

To prepare the secondary control mix equal volumes of the 0.05 mg/mL Secondary Standard Solution and the 0.1 mg/mL ISS.

#### 8.4.2.3. **Negative Control**

To prepare the negative control mix equal volumes of MeOH and the 0.1 mg/mL ISS.

8.4.2.4. Positive, secondary, and negative controls must be prepared from the same batch of ISS.



8.4.3. 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.

8.4.3.1. The instrument will be tuned weekly when in use or more frequently as deemed necessary. The tune will be evaluated according to established criteria for a successful tune as noted in the **Ideal Standard Tune** documents.

8.4.3.2. Printed copies of tune records are maintained in the section. If a successful tune cannot be achieved, the instrument will be taken out of service until instrument maintenance is performed. Documentation of the problem will be maintained in the section.

8.4.3.3. Although the monitoring of environmental ions such as water, oxygen, and nitrogen are included in the **Ideal Standard Tune** documents, the monitoring of these ions serves as a preventive measure because these ions are a guide to determining if there are leaks and/or water present in the system that could shorten the life of the column and/or the filaments. The presence of these ions does not prevent a tune from being deemed acceptable.

8.4.4. Solvent blanks will be injected immediately prior to and after all other injections to verify that the column and syringe are free of contamination. The solvent blanks will be run on the same method as the standard or sample runs.

8.4.5. Any significant peaks in the blank chromatograms will be properly investigated to identify their source (e.g. column breakdown, vial septa bleed, carryover from previous sample run, or instrumental contamination) so that appropriate action (such as replacing solvents or performing instrument maintenance) can be taken as necessary. Any affected case samples and associated blanks should be rerun (this is not necessary in the case of minor peaks identified as column breakdown or vial septa bleed).

## 8.5. Procedure

### 8.5.1. Preparation of Plant Substance Extract

8.5.1.1. Using an analytical balance, weigh out  $50 \text{ mg} \pm 0.5 \text{ mg}$  (0.0495 to 0.0505 g) of plant substance. The plant substance may be broken up manually if necessary.

8.5.1.2. Transfer plant substance to a culture tube.

8.5.1.3. Using a volumetric pipette or dispenser add 10mL of MeOH to the sample.

8.5.1.4. Vortex for 10 seconds, allow the sample to stand for 5 minutes, and vortex again for an additional 10 seconds.



8.5.1.5. Using a volumetric pipette mix equal volumes of the plant substance extract with the 0.1 mg/mL ISS in an autosampler vial for analysis by GC/MS. The sample must be prepared using the same batch of ISS as the controls.

8.5.1.6. Document the weight of plant substance and the pipette and/or dispenser used in the case record.

#### 8.5.2. GC/MS Operating Conditions

8.5.2.1. A GC/MS method has been developed as part of the decision-point assay that utilizes both the full scan and selected ion monitoring (SIM) modes for data acquisition. The full scan data produces a traditional mass spectrum which can be used for qualitative identification of substances in a sample. The SIM mode collects data for delta-9-THC and the internal standard and is used in determining if the concentration is above (greater than) or below (less than) the 1% decision-point.

8.5.2.2. For delta-9-THC the target SIM ion is 314 and the two qualifier ions are 231 and 271. For the internal standard (delta-9-THC-D3) the target SIM ion is 317 and two qualifier ions are 234 and 274 as these are the corresponding ions in the deuterated isomer.

8.5.2.3. Method parameters are available by the instrument or are electronically retrievable.

#### 8.5.3. Sample Analysis

8.5.3.1. Sample extracts and controls will be analyzed by injecting onto the GC/MS in the following order:

- Positive Control
- Secondary Control
- Negative Control
- Plant extracts
- Positive Control (reinjecting at the end of the batch)

8.5.3.2. The positive control will be reinjected after every ten sample extracts.

### 8.6. Interpretation

8.6.1. The results for the negative control will be examined to ensure that the ISS is free from contamination. If contamination is indicated, the cause will be investigated to determine if it originated from the instrument or the solution itself. If contamination appears to have originated from the instrument, then maintenance should be performed which may include injecting solvent blanks or inlet cleaning and the negative control rerun. If



contamination appears to have originated with the ISS, then the ISS should be discarded along with all other controls and extracts prepared from it.

- 8.6.2. Retention times and qualifier ion ratios for delta-9-THC and internal standard from the initial positive control injection will be used to set acceptance criteria. Retention times of delta-9-THC and internal standard for all subsequent controls within a batch shall be within 1% and qualifier ion ratios shall be within  $\pm 20\%$  of the established values for the initial positive control for the batch to be acceptable.
- 8.6.3. The relative peak area response (RPA, ratio of the abundance of delta-9-THC 314 ion and internal standard 317 ion) is determined for each control within a batch, and the average RPA is determined from these values. The individual RPA for all controls must be within 20% of the average RPA for the batch to be acceptable.
- 8.6.4. If the acceptance criteria for the controls within a batch are not met, then appropriate action must be taken which may include instrument maintenance, remaking the controls, or remaking the standard solutions. The sample extracts run within the batch will also need to be rerun. If an issue is identified with the ISS, then it will be remade, and all controls and sample extracts will be remade.
- 8.6.5. If the acceptance criteria for the controls within a batch are met, then the sample extract runs will be evaluated. For delta-9-THC to be identified within a run, the 314 target ion and both 231 and 271 qualifier ions must be detected. In addition, the retention times for delta-9-THC and the internal standard shall be within 1% and the qualifier ion ratios shall be within  $\pm 20\%$  of the established values for the initial positive control.
- 8.6.6. The RPAs for the initial and reinjected positive controls are compared, and the positive control with the highest RPA is used to establish a decision-point ratio (DPR) by normalizing all of the sample extract RPA values within the batch to this value. The positive control with the highest RPA will therefore always have a DPR value of 1.0 which corresponds to the 1% decision-point threshold.
- 8.6.7. Sample extracts that meet acceptance criteria with a DPR value at or above 1.0 met the administrative threshold for the identification of marihuana. See section 3.7 for further requirements for marihuana identification.
- 8.6.8. Sample extracts with a DPR value below 1.0 (or when acceptance criteria for the extract run are not met) do not meet the administrative threshold for the identification of marihuana. See section 3.7 for requirements for identification as Cannabis sativa L. in these cases.



8.6.9. The full scan data for sample extract runs may also be evaluated for the presence of additional compounds. See section 7.5.4 for analysis and interpretation of GC/MS data.

8.6.10. Data printouts for each sample extract run, batch controls, and the corresponding solvent blank runs will be labeled with the unique case identifier, item designators, date, and analyst's handwritten initials and will be maintained with the case file.

### **8.7. Extract Dilution**

8.7.1. When sample extracts do not produce results that meet acceptance criteria but have a DPR value above 1.0, then it is acceptable to perform an extract dilution and reanalyze. High concentrations of delta-9-THC in some samples can lead to unacceptable results and diluting these samples may yield acceptable results.

8.7.2. To prepare the extract dilution combine 1 part of sample extract with 9 parts of methanol. For example, using a volumetric pipette mix 50  $\mu$ l of sample extract with 450  $\mu$ l of methanol.

8.7.3. Using a volumetric pipette, the extract dilution is then mixed with equal volumes of the 0.1 mg/mL ISS in an autosampler vial for analysis by GC/MS. The sample must be prepared using the same batch of ISS as the controls.

### **8.8. Advantages**

8.8.1. Generally, mass spectra of compounds of interest are specific to single compounds and may be used for positive structural identification.

8.8.2. Use of SIM-scan mode allows for qualitative identification of compounds and evaluation of their concentration as being above or below a decision-point threshold from the same set of data.

8.8.3. Use of Gas Chromatography facilitates the determination of total delta-9-THC concentration (delta-9-THCA + delta-9-THC) as delta-9-THCA will be converted into delta-9-THC in the injector port.

8.8.4. Use of a 1% administrative decision-point threshold helps mitigate the risk of false positive identification of a substance as marijuana when it is not.

### **8.9. Limitations**

8.9.1. The decision-point assay is currently only applicable to plant substance.

8.9.2. Use of a 1% administrative decision-point threshold can result in samples which meet the statutory threshold of more than 0.3% delta-9-THC not being reported as marijuana.



8.9.3. Incomplete conversion of delta-9-THCA into delta-9-THC in the GC injector port may lead to sample results being below the 1% administrative decision-point threshold and therefore not being reported as marijuana.

8.9.4. High concentrations of cannabidiol (CBD) in plant substance samples can cause the results for delta-9-THC to be unacceptable and therefore lead to a sample not being reported as marijuana.

8.9.5. Plant substance samples may not be homogeneous in the amount of delta-9-THC which can lead to variability in results from different samples.

#### **8.10. Related Documents**

8.10.1. Instrument Logbook

8.10.2. Ideal Standard Tune for Agilent 6, 7, and 8



## 9. Fourier Transform Infrared (FTIR) Spectrometry

### 9.1. Scope

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

### 9.2. Safety

9.2.1. Use appropriate eye protection, gloves and lab coat when using solvents or chemicals. Refer to the SDSs for additional safety information for specific chemicals.

9.2.2. Discard all chemicals and any other pertinent materials in an appropriate manner.

### 9.3. Equipment, Materials, and Reagents

9.3.1. Fourier transform infrared spectrometer

9.3.2. Mortar and pestle (if needed)

9.3.3. Attenuated Total Reflectance (ATR) accessory

9.3.4. Acetone or suitable solvent (for cleaning)

### 9.4. Standards, Controls, and Calibration

9.4.1. A performance verification check will be performed quarterly or more often as needed and the results will be maintained in the section. One method is to use the OMNIC ValPro software to check the performance of the instrument. The measurements are made by ValPro utilizing a NG11 Glass Serialized Linearity standard and a 1.5 mil Serialized Polystyrene standard. ValPro tests the spectrophotometer's single-beam energy ratio, noise level, wavenumber accuracy, optical resolution, repeatability and detector linearity. A qualification report is provided to demonstrate the pass-fail results for each test.

9.4.2. If the report obtained from a performance verification check indicates failure of one or more tests, consult the FT-IR Operation Troubleshooting section of the FT-IR Spectrometer Validation handbook for potential causes and corrective recommendations. If these do not correct the problem, the instrument will be taken out of service until instrument maintenance is performed. Documentation of the problem will be maintained in the section.

9.4.3. The test results obtained by utilizing the ValPro performance checks are compared to prior results to verify that the system is working consistently over time.

9.4.4. A background will be taken before each sample scan and this step is included in the experimental method used for sample analysis.





## 9.5. Procedure

### 9.5.1. Sample Preparation

9.5.1.1. 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.

9.5.1.2. The sample must be in intimate contact with the ATR accessory sampling area to provide the highest signal. Methods of maximizing contact between the sample and sampling area include the following:

9.5.1.2.1. For liquid sampling, a trough insert is placed on the top of the ATR sampling plate and fastened with the knurled mounting ring. The insert forms a shallow well around the ATR crystal face for containment of the liquid. For routine liquids, place a drop of sample in the trough insert and collect data. For volatile liquids, the volatiles cover may be placed over the sample area to minimize evaporation of the sample.

9.5.1.2.2. Solid samples may be placed directly onto the surface of the crystal (with or without the trough). Since the ATR effect only takes place very close to the surface of the crystal, an intimate contact has to be made by the sample on the ATR crystal surface. This is achieved by using the pressure clamp. With the sample in place on the crystal, lower the pressure tip by turning the control knob so that it is in contact with the sample. Continue lowering the tip until the clamp clutch clicks.

### 9.5.2. Operating Conditions

Spectra are generally collected and printed with a resolution of at least  $4\text{ cm}^{-1}$  scanned from  $4000\text{ cm}^{-1}$  to  $600\text{ cm}^{-1}$  versus absorbance. This allows comparison to libraries and literature references with the same format.

### 9.5.3. Analysis and Interpretation

9.5.3.1. Sample spectra will be evaluated, printed, and included in the case file. The printouts will be labeled with the unique case identifier, item designators, and the analyst's handwritten initials. Spectra or notes will also include the date of observation and the method of sample preparation (if not listed on the **Examination Sheet**).

9.5.3.2. Spectra for each sample will be evaluated for suitability of comparison by first noting the amount of noise in the baseline. If the baseline is not smooth and shows



excessive variation or spikes, then the sample may need to be re-run. The presence of water and CO<sub>2</sub> will be observed as excessive amounts of either may require that the sample be re-run. The location, intensity, and shape of peaks will also be evaluated. Peaks that are too broad or too intense (flat at the top) may mean that too much sample was used and should be re-run. If peaks are too weak and difficult to distinguish from the baseline, then the sample should be re-run.

9.5.3.2.1. The analyst will determine identifications by comparing the unknown spectral peaks with those of known standards or published spectral data. The source for the comparison standard spectra, typically a stored library or a literature source, will be documented in the case file. 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 analyst verification of the overall appearance and the presence and location of major spectral peaks when making an identification.

9.5.3.2.2. If sample spectra do not have an acceptable reference comparison, then the printout will be labeled as "NAM" (for No Acceptable Match).

9.5.3.2.3. If the subtraction function is used to remove interfering substances, then retain a copy of the original sample spectrum with the case file. Also note the substances subtracted to generate the resulting spectrum.

9.5.3.2.4. If the straight-line function is used to remove interfering peaks from CO<sub>2</sub>, then retain a copy of the original spectrum with the case file. Also note the range over which the straight-line function was used.

## **9.6. Limitations**

9.6.1. When analysis by FTIR is unable to provide positive identification, another technique such as GC/MS must be utilized to provide positive identification.

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

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

9.6.4. Infrared spectroscopy cannot usually be used to distinguish between optical isomers.



### **9.7. Advantages**

- 9.7.1. Generally, infrared spectra of compounds of interest are specific to single compounds and may be used for positive structural identification.
- 9.7.2. Infrared is normally not a destructive test and the sample can be recovered for additional testing procedures, if necessary.
- 9.7.3. 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.

### **9.8. Literature and Supporting Documentation**

- 9.8.1. *FT-IR Spectrometer Validation*, Thermo Nicolet Corp., Madison WI, 2001.
- 9.8.2. "Standard Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests," ASTM E 1421-99, 1999.
- 9.8.3. Fell, A. F., *Clarke's Isolation and Identification of Drugs*, (London: The Pharmaceutical Society of Great Britain, 1986).
- 9.8.4. *Forensic Science Handbook*, Volume III, ed. By Richard Saferstein, (Englewood Cliffs, N.J.: Regents/Prentice Hall, 1993).
- 9.8.5. Skoog, D. A., *Principles of Instrumental Analysis*, 3<sup>rd</sup> Edition, (New York: Saunders College Publishing, 1985) 148-149.

### **9.9. Related Documents**

- 9.9.1. Instrument Logbook
- 9.9.2. Examination Sheet



## 10. Ultraviolet/Visible Spectrophotometry (UV/VIS)

### 10.1. Scope

10.1.1. An instrumental analytical technique for the screening of suspected controlled substances, dangerous drugs and other substances.

### 10.2. Safety

10.2.1. Use appropriate eye protection, gloves and lab coat when using acids, bases, or solvents to prepare solutions. Refer to the SDSs for additional safety information for specific chemicals.

10.2.2. Dispose of all chemicals in an appropriate manner.

### 10.3. Equipment, Materials, and Reagents

10.3.1. UV/VIS spectrophotometer

10.3.2. Quartz cuvettes, matched pair, or equivalent

10.3.3. An appropriate solvent for the sample

10.3.3.1. Acidic solutions, such as  $\frac{2}{3}$  N H<sub>2</sub>SO<sub>4</sub>

10.3.3.2. Basic solutions, such as 0.45 N NaOH

10.3.3.3. Methanol or ethanol

### 10.4. Standards, Controls, and Calibration

10.4.1. A UV/VIS performance verification check should be performed quarterly or as needed and the results will be maintained in the section. Check the wavelength accuracy using the two characteristic wavelength peaks of deuterium light at 486.0 nm and 656.1 nm. Follow the manufacturer's specifications for performing this check. The peak wavelength ranges should be between 485.5 nm - 486.5 nm and 655.6 nm - 656.6 nm respectively.

10.4.2. For comparison purposes, refer to reliable published reference materials, analyze known samples, or refer to in-house spectral collections produced from known samples.

10.4.3. Reference solvent blanks should be run at the same time using the same solvent as the sample.

10.4.4. If an instrument fails a performance check or a performance problem is detected during routine maintenance or use, it will be taken out of service until instrument maintenance is performed. Documentation of the problem will be maintained in the section.



## 10.5. Procedure

### 10.5.1. Sample Preparation

10.5.1.1. Dissolve the sample in a solvent/solution appropriate for the substance.

10.5.1.2. Plant materials will require extraction, while mixtures and other substances may require extraction prior to analysis.

### 10.5.2. Operating Conditions

10.5.2.1. The wavelength range used for the UV/VIS analysis of most drug samples is 340 to 220 nm, but may need to be expanded to accommodate certain substances such as alkyl nitrites, GHB, and GBL.

10.5.2.2. Depending on the concentration of the sample, it may be necessary to dilute the solution so that the absorbance range is between 0 - 2 units.

10.5.2.3. A "pH shift" may be performed on basic drugs in acidic solutions by adding an appropriate base until the solution is basic. For acidic drugs the process is reversed.

### 10.5.3. Analysis and Interpretation

10.5.3.1. Sample spectra will be evaluated, printed, and included in the case file. The printouts will be labeled with the unique case identifier, item designators, and the analyst's handwritten initials. Spectra or notes will also include the date of observation, and the method of sample preparation (if not listed on the **Examination Sheet**).

10.5.3.2. Spectra for each sample will be evaluated for suitability of comparison by first noting the amount of noise in the baseline. If the baseline is not smooth and shows excessive variation or spikes, then the sample may need to be re-run. The complete absence of absorption bands may indicate that another solvent/solution or scan range needs to be used. The location, intensity, and shape of absorption bands will also be evaluated. If bands are too intense (flat on the top) or too weak and difficult to distinguish from the baseline, then the sample should be re-run.

10.5.3.2.1. The analyst will compare acceptable sample spectra with documented reference sources or spectra from known drug standards.

10.5.3.2.2. If sample spectra do not have an acceptable reference comparison, then the printout will be labeled as "NAM" (for No Acceptable Match).



### **10.6. Limitations**

- 10.6.1. The results of UV/VIS analysis are not considered to be specific in nature and further structural confirmation by instrumental analysis is necessary for the positive identification of a questioned substance.
  
- 10.6.2. Not all substances absorb ultraviolet light; therefore, the lack of absorbance or a flat-line spectrum is not necessarily an indication that a sample does not contain a controlled substance or dangerous drug (e.g. carisoprodol has no UV absorption from 220 – 340 nm).
  
- 10.6.3. 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.

### **10.7. Advantages**

- 10.7.1. The test is quick and easy to perform.
  
- 10.7.2. Usually very little sample preparation is required.
  
- 10.7.3. 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 (diluent) and adulterants.
  
- 10.7.4. This is usually a non-destructive technique and the sample can be recovered for other testing procedures, if necessary.

### **10.8. Literature and Supporting Documentation**

- 10.8.1. Sandor Gorog, *Ultraviolet-Visible Spectrophotometry in Pharmaceutical Analysis* (CRC Press, 1995).
  
- 10.8.2. A. F. Fell, "Ultraviolet, Visible, and Fluorescence Spectrophotometry", *Clarke's Isolation and Identification of Drugs*, Second Edition, (London: The Pharmaceutical Press, 1986), 221-236.
  
- 10.8.3. A.C. Moffat, et. al., "Ultraviolet, Visible, and Fluorescence Spectrophotometry", *Clarke's Analysis of Drugs and Poisons*, Third Edition, (London: The Pharmaceutical Press, 2004), 313-327.
  
- 10.8.4. Douglas A. Skoog and Donald M. West, *Principles of Instrumental Analysis* (New York: Holt, Rinhart, and Winston, Inc., 1971).
  
- 10.8.5. Terry Mills III and Conrad J. Roberson, *Instrumental Data for Drug Analysis*, (New York: Elsevier Science Publishing Co., Inc., 1987).



**10.9. Related Documents**

10.9.1. Instrument Logbook

10.9.2. Examination Sheet



## 11. Drug Standards and Reference Sources

### 11.1. Scope

11.1.1. These policies serve to establish guidelines for the use of drug standards, comparison sources, and libraries.

### 11.2. Quality Control Procedures for Drug Standards

11.2.1. Drug standards available for use in the Seized Drugs section may be purchased from commercial vendors, received from another forensic laboratory, or obtained from properly characterized casework samples.

11.2.2. Before using any new drug standard regardless of its source, an FTIR or GC/MS will be performed to verify that the compound is what it is purported to be. This requirement includes plant substance samples. The verification will be documented as part of the **Drug Standard Verification Log** which will include the name of the drug standard, common names, in-house identification number, location (if stored somewhere other than the designated locked cabinet such as a secured refrigerator), source and lot number, type of verification and date, expiration date if applicable, and final disposition. The spectra obtained for verification will be placed in a quality control book which will include all pertinent information such as the standard name, identification number, the initials of the analyst who performed the test, the date, and comparison data.

11.2.3. Drug standards available in the Seized Drugs section will be documented on a **Drug Standard Usage Log** which will include the name of the drug standard, the in-house identification number, and the lot number. This sheet will also be used to document when significant quantities of the drug standard are used, by whom, and the reason for use (training, performance checks, etc.).

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

11.2.5. The use of casework samples as drug standards will be documented in the originating case record.

11.2.6. Drug standards will be stored in a securely locked cabinet or refrigerator that can be accessed only by persons authorized by the section manager. Aliquots or small portions of





these drug standards may be prepared and kept in the locked cabinet or refrigerator or at an analyst's work area for quality checks or use in routine analysis such as TLC.

11.2.7. An annual inventory of the available drug standards will be conducted and recorded on the **Drug Standard Usage Log** in conjunction with the **Drug Standard Verification Log**. This inventory will document those standards that have been consumed and need to be replaced as well as those standards that have expired and need to be re-verified or discarded. Standards that have been discarded or consumed will be identified on both the **Drug Standard Usage Log** and the **Drug Standard Verification Log**.

### **11.3. Comparison Sources and Library References**

11.3.1. References used for pharmaceutical identification will be documented in the case file.

The following is a list of commonly used pharmaceutical references (other sources may be used as long as they are properly documented in the case file):

- 11.3.1.1. Physician's Desk Reference (PDR)
  - Amera-Chem Logo Search (ACLS)
  - DEA Logo Search (DEA)
  - Poison Control
  - Drug Identification Bible (DIB)
  - Drugs.com (<http://www.drugs.com>)
  - Pharmaceutical identification from packaging or manufacturer information

11.3.2. When analyzing compounds, particularly drugs, using either GC/MS or FTIR, the spectra will be compared to a standard from a reference source. The source of the standard spectrum will be documented in the case file. The following is a list of common reference sources for standard GC/MS and FTIR spectra (other sources may be used as long as they are properly documented in the case file):

- 11.3.2.1. NIST mass spectral library (various editions)
  - SWGDRUG mass spectral library
  - American Academy of Forensic Sciences (AAFS) mass spectral library
  - In-house mass spectral library
  - Georgia State Crime Lab FTIR library
  - In-house FTIR spectral library
  - Clarke's Isolation and Identification of Drugs (various editions)
  - Mills Instrumental Data for Drug Analysis (various editions)
  - CND Analytical series
  - Microgram Journal / Bulletin
  - Journal of Forensic Science



CLIC Journal  
Forensic Science International  
Forensic Toxicology

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

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

11.3.5. For reference libraries produced in-house, the spectral information for each library entry must be matched to information for the same compound that is published in an approved library or literature source. The person that performs the comparison must note, either on the reference spectrum itself or in the information that accompanies it, the source of the reference used for the comparison and his or her initials.

#### **11.4. Related Documents**

11.4.1. Drug Standard Usage Log

11.4.2. Drug Standard Verification Log



## **12. Reagent Quality Assurance**

### **12.1. Scope**

12.1.1. The following describes quality assurance guidelines for reagents and chemical preparations used in analysis.

### **12.2. Safety**

12.2.1. Use appropriate eye protection, gloves and lab coat to avoid contact with chemicals.

12.2.2. Refer to the appropriate SDSs for the safe handling of chemicals.

12.2.3. Discard all chemicals and any other pertinent materials in an appropriate manner.

### **12.3. Practice**

#### **12.3.1. Labeling**

12.3.1.1. All pertinent reagents and solutions will be labeled with the identity of the reagent, concentration (if applicable), and the date of preparation (or lot number), and, as applicable, storage requirements.

12.3.1.2. A Reagent Logbook will be maintained and will include the following information, when applicable:

12.3.1.2.1. Reagent preparation date

12.3.1.2.2. Preparer's initials

12.3.1.2.3. Standard used and the results of a positive quality control check of the reagent

12.3.1.2.4. Results of a negative (blank) quality control check of the reagent

12.3.1.2.5. Initials of the analyst(s) who quality tested the reagent and the date of testing



### 12.3.2. Quality Testing for Frequently Used Reagents

12.3.2.1. **Frequently** used reagents will be quality tested prior to their initial use and monthly thereafter. Upon preparation, the preparer will record his or her initials in the logbook along with the date prepared. This same date will also be reflected on the stock reagent container. When the reagent is quality tested the appropriate information is recorded in the logbook. The quality testing will include both a positive control using an appropriate standard and a negative (blank) control. In addition to the date of preparation, the date of the most recent quality test will be noted on the stock reagent bottle.

12.3.2.2. All general use containers (aliquots) of frequently used reagents will be quality tested monthly along with the stock reagent and the results recorded in the logbook. These containers will be labeled with the date of reagent preparation and the date of the most recent quality test. When a new stock reagent is prepared, the general use containers will be replaced with this reagent after it has been quality checked.

12.3.2.3. Aliquots for frequently used reagents at an analyst's work area will be replaced each month from the stock reagent bottle after it has been quality checked. These containers will be labeled with the date of reagent preparation and the date of the most recent quality test. It is the analyst's responsibility to document replacement of his/her aliquots.

12.3.2.4. See the Chemical Spot Tests section for a listing of the current **Frequently** used reagents.

### 12.3.3. Quality Testing for Infrequently Used Reagents

12.3.3.1. **Infrequently** used reagents will be quality tested prior to their initial use and the results as well as the preparer's initials and the date of preparation will be recorded in the logbook. Subsequent quality testing will be performed by the analyst prior to use and the results as well as the standard used will be documented in the case notes.

12.3.3.2. Aliquots for infrequently used reagents at an analyst's work area will be labeled with the date of reagent preparation.



#### 12.3.4. Quality Testing for TLC Reagents

12.3.4.1. Upon preparation, **TLC** (thin layer chromatography) reagents will be documented in the logbook with the date prepared and the preparer's initials. TLC reagents will be quality tested during use by the analyst using an appropriate standard and the results will be documented in the case notes.

#### 12.3.5. Quality Testing for Acids and Bases

12.3.5.1. Upon preparation, **acidic and basic** solutions will be documented in the logbook with the date prepared, the preparer's initials, and the results of a pH check.

12.3.5.2. Aliquots for acidic and basic solutions at an analyst's work area will be labeled with the date of preparation.

#### 12.3.6. Quality Assurance

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

12.3.6.2. If an analyst has reason to suspect that a reagent or other chemical preparation is not working properly or is contaminated, he or she must:

12.3.6.2.1. Cease performing casework with these reagents until the problem has been corrected.

12.3.6.2.2. Check the reagent or system with standards or proper sample controls.

12.3.6.2.3. Discard the reagent if it fails the quality check, prepare a new reagent, and quality check the new reagent with a known standard.

12.3.6.2.4. Identify casework that may have been affected by the reagents/chemicals that failed the quality check and re-test with quality checked reagents.

12.3.6.2.5. Inform the section manager and Quality director if the problem persists.



#### **12.4. Related Documents**

12.4.1. Reagent Logbook

12.4.2. Monthly Quality Check for Frequently Used Chemical Spot Test Reagents

12.4.3. Monthly Quality Check for Frequently Used Stock Reagents

12.4.4. Monthly Quality Check for General Frequently Used Reagent Aliquots

12.4.5. Quality Check for Infrequently Used Stock Reagents

12.4.6. Quality Check for Stock Acids and Bases

12.4.7. Quality Check for TLC Solvent and Indicator Sprays



### 13. Chemical Spot Tests

#### 13.1. Scope

13.1.1. To describe the chemical screening procedures commonly referred to as color tests or spot tests for the analysis of suspected controlled substances, dangerous drugs and other substances.

#### 13.2. Safety

13.2.1. Chemical spot tests may use a variety of corrosive, caustic, or other dangerous chemicals. Caution should always be practiced, and eye protection, gloves and lab coat should be used.

13.2.2. Refer to SDSs for additional safety information for specific chemicals.

13.2.3. Discard all chemicals, reagents, and any other pertinent materials in an appropriate manner.

#### 13.3. Equipment, Materials, and Reagents

13.3.1. Spot plates, pipettes, or other appropriate containers/items.

13.3.2. Reagents appropriate to the specific chemical spot tests.

#### 13.4. Standards and Controls

13.4.1. Each spot test stock reagent must be labeled with the name of the reagent, concentration (if applicable), as well as the date of preparation (or lot number). A quality control log book will be maintained and will include the preparer's initials and the date prepared as well as the results of appropriate quality testing.

13.4.2. The frequently used spot test reagents are Ferricyanide, Marquis, Van Urk's, Cobalt thiocyanate, and Duquenois. These reagents will be quality tested prior to their initial use and monthly thereafter with the date of preparation and most recent quality testing noted on all in use containers. All other spot test reagents are considered infrequently used and must be quality checked prior to their initial use and again by the analyst prior to use for casework samples.

13.4.3. It is the responsibility of the analyst to quality check infrequently used reagents prior to use and document appropriately on the **Examination Sheet**. Proper documentation



includes noting the reagent used, the standard used, and the results. See the Reagent Quality Assurance section for further explanation of quality testing procedures.

13.4.4. It is the responsibility of the analyst to determine if reagents are working properly prior to use. Blank (or negative) controls for chemical spot tests are to be performed at the same time as sample testing to demonstrate that the reagents used are not contaminated. If the blank control shows a positive reaction (is not negative), then the reagents will be discarded and replaced with fresh quality tested aliquots. In addition, spot plates used to perform chemical spot tests are to be visually examined by the analyst prior to use to ensure that they are free of debris or residue. If a spot plate is not clean, then it will not be used for analysis. These checks will be documented on the **Examination Sheet**.

### **13.5. Definitions**

13.5.1. Purified water means water that is purified by either deionization or distillation. All water used to prepare spot test reagents will be purified water.

### **13.6. Interpretation**

13.6.1. Any reaction observed by the analyst will be documented on the **Examination Sheet** by writing the color observed.

13.6.2. With weak color changes, the analyst may choose to document the color preceded by the designation "weak."

13.6.3. The remainder of this section includes spot tests commonly used in the Seized Drugs section, recipes for preparation, procedures for use, and interpretation of results. The examples of listed interpretations are not intended to be an exhaustive list of all possibilities. Comparison of the results obtained from samples with standards and documentation of the results is considered to be sufficient for additional interpretations.

### **13.7. Limitations**

13.7.1. The results of spot tests are not considered to be specific in nature and further structural confirmation by instrumental analysis is necessary for the positive identification of a questioned substance.

13.7.2. Adulterants and complex mixtures may produce reactions that interfere with the clear interpretation of the results.





13.7.3. A sample with a low concentration of a particular substance may yield negative (no color reaction observed) spot test results.

### **13.8. Advantages**

13.8.1. Spot tests provide a quick and easy method for determining what type of compound or functional group a sample might contain.

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

### **13.9. Related Documents**

13.9.1. Reagent Logbook

13.9.2. Examination Sheet



### 13.10. Koppanyi Test

#### 13.10.1. Reagents/Chemicals

- Cobalt nitrate,  $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$
- Isopropylamine
- Methanol

*1% Cobalt Nitrate Reagent:* Dissolve 8.0 g  $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$  in 500 ml methanol.

*5% Isopropylamine Reagent:* Add 5 ml isopropylamine to 95 ml methanol.  
(Stock reagent stored in the refrigerator).

Quality-test reagent with a barbiturate standard.

#### 13.10.2. Procedure

- 13.10.2.1. Combine a small amount of sample and a few drops of 1% cobalt nitrate reagent.
- 13.10.2.2. Record any observations.
- 13.10.2.3. Add a few drops 5% isopropylamine reagent to sample.
- 13.10.2.4. Record any observations.

#### 13.10.3. Interpretation

- 13.10.3.1. Formation of a purple color upon addition of the 1% cobalt nitrate reagent indicates the possible presence of gamma-hydroxybutyrate (GHB).
- 13.10.3.2. A few of the barbiturates will form a purple color with the addition of the first reagent.
- 13.10.3.3. Formation of a purple color which forms after the addition of the 5% isopropylamine reagent indicates the possible presence of barbiturates.
- 13.10.3.4. Sometimes vitamin C, ibuprofen, and lactose fillers in tablets will exhibit a faint purple color.



#### 13.10.4. Literature and Supporting Documentation

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

13.10.4.2. W.J. Stall, "The Cobalt Nitrate Color Test," *Microgram* 13(3), 1980, pp. 40-43.

13.10.4.3. J.A. Morris, "Extraction of GHB for FTIR Analysis and a New Color Test for Gamma-Butyrolactone (GBL)," *Microgram* 32(8), 1999, pp. 215-221.



### 13.11. Ferricyanide Test (also known as Simon's test)

#### 13.11.1. Reagents/Chemicals

- Sodium nitroferricyanide (sodium nitroprusside)
- Acetaldehyde
- Purified water
- 20% Sodium carbonate

*Ferricyanide Reagent:* Dissolve 4 g sodium nitroferricyanide in a mixture of 40 ml acetaldehyde and 400 ml water. (Stock reagent stored in the refrigerator)

Quality-test reagent with a methamphetamine standard.

#### 13.11.2. Procedure

- 13.11.2.1. Combine a small amount of sample with a few drops of ferricyanide reagent.
- 13.11.2.2. Add a few drops of 20% sodium carbonate.
- 13.11.2.3. Record any observations.
- 13.11.2.4. The reagent combination itself turns a deep red. This color is the normal color for a negative reaction.

#### 13.11.3. Interpretation

- 13.11.3.1. Formation of a blue color with the addition of the 20% sodium carbonate indicates the possible presence of secondary amines (e.g. MDMA, methamphetamine, methylphenidate, BZP, TFMPP).
- 13.11.3.2. Some secondary amines (MDE, N-OH MDA) do not form a blue color or form only a slight purple color due to steric hindrance.
- 13.11.3.3. Strongly basic solutions will form a deep red color before the addition of the 20% sodium carbonate.

#### 13.11.4. Literature and Supporting Documentation



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



## 13.12. Marquis Test

### 13.12.1. Reagents/Chemicals

- Concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ )
- Formaldehyde solution (~ 37% formaldehyde)

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

### 13.12.2. Procedure

13.12.2.1. Combine a small amount of sample with a few drops of concentrated  $\text{H}_2\text{SO}_4$ .

13.12.2.2. Add one drop of formaldehyde solution.

13.12.2.3. Record any resulting color reactions. Generally, color reactions are observed after the addition of the formaldehyde solution, but for certain substances color changes may occur with the initial addition of concentrated  $\text{H}_2\text{SO}_4$ . When this occurs a slash mark may be used to document color reactions that are observed in the first step and then in the second step (for example "yellow/yellow" or "purple/yellow").

### 13.12.3. Interpretation

13.12.3.1. Formation of an orange to brown color indicates the possible presence of amphetamine, methamphetamine or phentermine (other substances may show similar color formations).

13.12.3.2. Formation of an orange (sometimes orange to brown) color indicates the possible presence of fentanyl or fentanyl derivatives.

13.12.3.3. Formation of a purple to black color indicates the possible presence of MDMA, MDE, and MDA.

13.12.3.4. Formation of a green to black color indicates the possible presence of dextromethorphan.

13.12.3.5. Formation of a green color indicates the possible presence of 2,5-dimethoxyphenethylamine and its derivative 4-bromo-2,5-dimethoxyphenethylamine (Nexus, 2C-B).



- 13.12.3.6. Formation of a purple color indicates the possible presence of heroin, other opiates, methocarbamol, or guaifenesin.
- 13.12.3.7. Formation of a red color indicates the possible presence of salicylates (Aspirin).
- 13.12.3.8. Formation of a dark red color indicates the possible presence of toluene.
- 13.12.3.9. Some benzodiazepines such as diazepam form an orange color after several minutes.
- 13.12.3.10. Formation of a yellow color with the concentrated acid that persists with the addition of the formaldehyde solution indicates the possible presence of diphenhydramine or methylenedioxy cathinones such as methylone, butylone, pentylone, or MDPV.
- 13.12.3.11. A yellow powder which forms a deep purple color with the addition of the concentrated acid followed by a change to yellow with the addition of the formaldehyde solution indicates the possible presence of tetracycline.
- 13.12.3.12. Formation of a black color upon the addition of the concentrated acid then orange with fizzing upon the addition of the formaldehyde solution (due to the release of NO<sub>2</sub>) indicates the possible presence of a nitrite.
- 13.12.3.13. There may be other substances that form various colors with the reagents.

#### 13.12.4. Literature and Supporting Documentation

- 13.12.4.1. H.M. Stevens 1986. "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A.C. Moffat (London: The Pharmaceutical Press) pp. 128-147.
- 13.12.4.2. S.H. Johns, et. al. "Spot Tests: A Color Chart Reference for Forensic Chemists," *Journal of Forensic Sciences* 24 (1979) pp. 631-649.
- 13.12.4.3. F.T. Noggle, et. al. "Analytical Profiles of 4-bromo-2,5-dimethoxyphenethylamine ("Nexus") and Related Precursor Chemicals," *Microgram* 27(10), Oct. 1994, pp. 343-355.



13.12.4.4. K. E. Toole, et. al. "Color Tests for the Preliminary Identification of Methcathinone and Analogues of Methcathinone," *Microgram Journal* 9(1), pp. 27-32.





### 13.13. Van Urk's Test (also known as *p*-Dimethylaminobenzaldehyde or Erlich's Test)

#### 13.13.1. Reagents/Chemicals

- *p*-Dimethylaminobenzaldehyde (*p*-DMAB)
- 95% Ethanol
- Concentrated sulfuric acid

*Van Urk's Reagent*: Dissolve 4 g *p*-DMAB in 450 ml 95% ethanol. Very slowly add 50 ml concentrated sulfuric acid (Stock reagent stored in the refrigerator)

Quality-test reagent with benzocaine, procaine, or lysergic acid diethylamide.

#### 13.13.2. Procedure

- 13.13.2.1. Combine a small amount of sample and a few drops of Van Urk's reagent.
- 13.13.2.2. Record any observations.

#### 13.13.3. Interpretation

- 13.13.3.1. Formation of a bright yellow color indicates the possible presence of primary aromatic amines such as procaine and benzocaine.
- 13.13.3.2. Formation of a purple color indicates the possible presence of some indole containing compounds such as melatonin and 5-methoxy-N,N-diisopropyltryptamine (5-MeO-DIPT, and Foxy-Methoxy).
- 13.13.3.3. Formation of a purple color indicates the possible presence of LSD and some other ergot alkaloids (this reaction can take as long as five to ten minutes to occur).

#### 13.13.4. Literature and Supporting Documentation

- 13.13.4.1. H.M. Stevens, 1986. "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A.C. Moffat (London: The Pharmaceutical Press) pp. 128-147.
- 13.13.4.2. S.H. Johns, et. al. "Spot Tests: A Color Chart Reference for Forensic Chemists," *Journal of Forensic Sciences*, 24 (1979): pp. 631-649.



13.13.4.3. Basic Training for Forensic Drug Chemists, U.S. Dept. of Justice, 3<sup>rd</sup> edition.

13.13.4.4. T.K. Spratley, et. al. "Analytical Profiles for Five "Designer" Tryptamines,"  
*Microgram Journal* Vol. 3 (1-2), Jan-June 2005, pp. 54-68.



### 13.14. Cobalt Thiocyanate (Cocaine test; Scott's test)

#### 13.14.1. Reagents/Chemicals

- Cobalt thiocyanate
- 96% USP glycerine (glycerol)
- Purified water
- Concentrated hydrochloric acid
- Chloroform

*Cobalt thiocyanate Reagent:* Dissolve 2 g cobalt thiocyanate in 100 ml water and dilute with 100 ml glycerine.

Quality-test reagent with a cocaine standard.

#### 13.14.2. Procedure

- 13.14.2.1. Combine a small amount of sample with the cobalt thiocyanate reagent.
- 13.14.2.2. If a color change is observed, the analyst will record any observations and may stop at this step.
- 13.14.2.3. Add one drop of concentrated hydrochloric acid.
- 13.14.2.4. Add a few drops of chloroform to extract any soluble complexes.
- 13.14.2.5. Record any observations.

#### 13.14.3. Interpretation

- 13.14.3.1. The cobalt thiocyanate test is useful in distinguishing cocaine salt from cocaine base when all of the steps are performed.
- 13.14.3.2. If addition of the cobalt thiocyanate reagent results in the formation of a blue color which disappears upon addition of the concentrated HCl and reappears in the chloroform layer, then a cocaine salt could be present.
- 13.14.3.3. If addition of the cobalt thiocyanate reagent results in no color formation or a light blue color around the surface of the particles followed by a blue color with addition of concentrated HCl which transfers to the chloroform layer, then cocaine base could be present.



13.14.3.4. Some other substances that form a blue color with the addition of the cobalt thiocyanate reagent are acetone, lidocaine, PCP, heroin (if concentrated enough), gamma-butyrolactone, and diphenhydramine.

13.14.4. Literature and Supporting Documentation

13.14.4.1. L.J. Scott, "Specific Field Test for Cocaine, " *Microgram* 6 (1973): pp. 179-181.

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

13.14.4.3. A.L. Deakin, "A Study of Acids Used for the Acidified Cobalt Thiocyanate Test for Cocaine Base," *Microgram Journal* 1(1-2), Jan-June 2003, pp. 40-43.

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

13.14.4.5. J.A. Morris, "Extraction of GHB for FTIR Analysis and a New Color Test for Gamma-Butyrolactone (GBL)," *Microgram* 32(8), 1999, pp. 215-221.



### 13.15. Janovsky Test

#### 13.15.1. Reagents/Chemicals

- *m*-Dinitrobenzene
- 95% Ethanol
- Purified water
- Potassium hydroxide

*2% m-Dinitrobenzene Reagent:* Dissolve 4 g *m*-dinitrobenzene in 200 ml 95% ethanol.

*5 N Potassium Hydroxide:* Dissolve 56 g potassium hydroxide in 200 ml water.

Quality-test reagent with diazepam standard.

#### 13.15.2. Procedure

13.15.2.1. Combine a small amount of sample with equal parts of 2% *m*-dinitrobenzene reagent and 5 N potassium hydroxide.

13.15.2.2. Record any observations.

#### 13.15.3. Interpretation

13.15.3.1. Formation of a purple color indicates the possible presence of diazepam or flunitrazepam.

13.15.3.2. Some references have indicated that ketamine will form a blue color with the test, but our observations have been that the color formation is to purple and not consistent enough for reliability.

13.15.3.3. Formation of a yellow color indicates the possible presence of clonazepam or nitrazepam.

13.15.3.4. No color formation is seen with alprazolam or lorazepam.



#### 13.15.4. Literature and Supporting Documentation

- 13.15.4.1. C.L. Rucker, "Chemical Screening and Identification Techniques for Flunitrazepam," *Microgram* 31(7), 1998, pp. 198-205.



### 13.16. Weber Test

#### 13.16.1. Reagents/Chemicals

- Fast Blue B salt
- Concentrated hydrochloric acid
- Purified water

*0.1% Fast Blue B Reagent:* Dissolve 0.1 g Fast Blue B salt in 100 ml water.

Prepare this reagent fresh and quality-test with standard psilocin before use.

#### 13.16.2. Procedure

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

13.16.2.2. Record any observations.

13.16.2.3. Add a few drops of concentrated hydrochloric acid.

13.16.2.4. Record any observations.

#### 13.16.3. Interpretation

13.16.3.1. Formation of a red color with addition of the Fast Blue B reagent which changes to blue with the addition of the acid indicates the possible presence of psilocin.

#### 13.16.4. Literature and Supporting Documentation

13.16.4.1. A.S. Garrett, S.R. Clemons, J.H. Gaskill, "The Weber Test: A Color Test for the Presence of Psilocin in Mushrooms", *SWAFS Journal*, 15(1), April 1993, pp. 44-45.



### 13.17. Ferric Chloride Test

#### 13.17.1. Reagents/Chemicals

- Ferric chloride,  $\text{FeCl}_3 \bullet 6 \text{H}_2\text{O}$
- Purified water

*5% Ferric Chloride Reagent:* Dissolve 8.3 g  $\text{FeCl}_3 \bullet 6 \text{H}_2\text{O}$  in 100 ml water.

Quality-test with gamma-hydroxybutyric acid (GHB) standard.

#### 13.17.2. Procedure

13.17.2.1. Combine a small amount of sample with a few drops of 5% ferric chloride reagent.

13.17.2.2. Record any observations.

#### 13.17.3. Interpretation

13.17.3.1. Formation of a red-orange color indicates the possible presence of GHB.

13.17.3.2. Formation of a dark purple color indicates the possible presence of salicylates (aspirin).

13.17.3.3. Formation of a bluish gray color indicates the possible presence of acetaminophen.

#### 13.17.4. Literature and Supporting Documentation

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

13.17.4.2. J.A. Morris, "Extraction of GHB for FTIR Analysis and a New Color Test for Gamma-Butyrolactone (GBL)," *Microgram* 32(8), 1999, pp. 215-221.





### 13.18. Liebermann Test

#### 13.18.1. Reagents/Chemicals

- Sodium nitrite
- Concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)

*Liebermann's Reagent:* Carefully add 5 g sodium nitrite to 50 ml concentrated H<sub>2</sub>SO<sub>4</sub> with cooling and swirling. Perform the addition in the hood, as toxic nitrogen oxides are produced.

Quality-test the reagent with a standard of methylphenidate, ephedrine, mescaline, or dextropropoxyphene.

#### 13.18.2. Procedure

13.18.2.1. Combine a small amount of sample and a few drops of Liebermann's reagent.

13.18.2.2. Record any observations.

#### 13.18.3. Interpretation

13.18.3.1. Various colors may be formed by a large number of different compounds. Results or interpretations can be found in Stevens (1986).

13.18.3.2. A variety of color results for steroids may be found in Chiong (p.491).

#### 13.18.4. Literature and Supporting Documentation

13.18.4.1. H.M. Stevens, 1986: "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A.C. Moffat (London: The Pharmaceutical Press) pp. 127-147.

13.18.4.2. D.M. Chiong, E. Consuega-Rodriguez, and J.R. Almirall, "The Analysis and Identification of Steroids", *Journal of Forensic Sciences*, 37(2), March 1992, pp 488-502.



### 13.19. Sulfuric Acid Test

#### 13.19.1. Reagents/Chemicals

- Concentrated sulfuric acid

Quality-test reagent with a steroid standard.

#### 13.19.2. Procedure

13.19.2.1. Combine a small amount of sample and a few drops of concentrated sulfuric acid.

13.19.2.2. Record any observations. A UV light may be used to aid visualization of a color change.

#### 13.19.3. Interpretation

13.19.3.1. Formation of an orange or yellow color may indicate the possible presence of a steroid.

13.19.3.2. Formation of a yellow color may also indicate the possible presence of diphenhydramine or methylenedioxy cathinones such as methylone, butylone, pentylone, or MDPV. See **Marquis Test**.

#### 13.19.4. Literature and Supporting Documentation

13.19.4.1. H.M. Stevens, 1986: "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A. C. Moffat (London: The Pharmaceutical Press) pp. 127-147.

13.19.4.2. D.M. Chiong, E. Consuega-Rodriguez, and J.R. Almirall, "The Analysis and Identification of Steroids", *Journal of Forensic Sciences*, 37(2), March 1992, pp 488-502.



## 13.20. Mandelin Test

### 13.20.1. Reagents/Chemicals

- Ammonium vanadate
- Concentrated sulfuric acid
- Purified water

*Mandelin's Reagent:* Dissolve 0.5 g ammonium vanadate in 1.5 ml water. Carefully dilute to 100 ml with concentrated sulfuric acid. Filter the reagent through glass wool.

Quality-test with a codeine standard.

### 13.20.2. Procedure

- 13.20.2.1. Combine a small amount of sample and a few drops of Mandelin's reagent.
- 13.20.2.2. Record any observations.

### 13.20.3. Interpretation

- 13.20.3.1. Various colors may be produced by a large number of different compounds including codeine which is indicated by the formation of a green color. Results and interpretations may be found in Stevens (1986).
- 13.20.3.2. A variety of color changes for steroids may be found in Chiong (p. 491).

### 13.20.4. Literature and Supporting Documentation

- 13.20.4.1. H.M. Stevens, 1986: "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A. C. Moffat (London: The Pharmaceutical Press) pp. 127-147.
- 13.20.4.2. D.M. Chiong, E. Consuega-Rodriguez, and J.R. Almirall, "The Analysis and Identification of Steroids", *Journal of Forensic Sciences*, 37(2), March 1992, pp 488-502.



### 13.21. Duquenois-Levine Test

#### 13.21.1. Reagents/Chemicals

- Vanillin
- 95% Ethanol
- Acetaldehyde (Stored in the refrigerator)
- Concentrated hydrochloric acid
- Chloroform
- Petroleum ether

*Duquenois Reagent:* Add 19.2 g vanillin and 2.4 ml acetaldehyde to 960 ml 95% ethanol. (Stock reagent stored in the refrigerator)

Quality-test with a known *Cannabis sativa* L. or **marihuana** sample.

#### 13.21.2. Procedure

- 13.21.2.1. Place a small amount of material in a testing container. Either proceed to the next step or extract the material with a suitable solvent such as petroleum ether. If extracted, discard the material and evaporate to dryness.
- 13.21.2.2. Add one part of the Duquenois reagent and wait approximately one minute.
- 13.21.2.3. Add one part concentrated hydrochloric acid.
- 13.21.2.4. Record any observations.
- 13.21.2.5. Add one part chloroform (the Levine modification).
- 13.21.2.6. Record any observations.

#### 13.21.3. Interpretation

- 13.21.3.1. Formation of a purple color after the addition of concentrated hydrochloric acid to the mixture of Duquenois reagent and material or extract is a positive reaction and indicates the possible presence of cannabinoids, including tetrahydrocannabinol (THC).
- 13.21.3.2. Formation of a purple color in the chloroform layer indicates the possible presence of cannabinoids, including tetrahydrocannabinol (THC).



13.21.3.3. Formation of a purple color in both reactions above indicates that cannabinoids are present.

13.21.4. Literature and Supporting Documentation

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

13.21.4.2. K. Bailey, "The Value of the Duquenois Test for Cannabis – A Survey", *Journal of Forensic Science*, 24 (1979): pp. 817-841.



**14. Chemical Microcrystalline Tests (Rescinded as of December 1, 2016)**



## 15. Thin Layer Chromatography (TLC)

### 15.1. Scope

15.1.1. To describe the screening procedure commonly referred to as thin-layer chromatography, for the analysis of suspected controlled substances, dangerous drugs and other substances.

### 15.2. Safety

15.2.1. 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.

15.2.2. 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 SDSs for the safe handling of the solvents and reagents used in this technique.

15.2.3. Developing solvent systems and indicator reagents should be discarded in an appropriate manner.

### 15.3. Equipment, Materials, and Reagents

15.3.1. TLC plates with silica gel on aluminum, glass, polyester, or other appropriate medium

15.3.2. Glass developing tank

15.3.3. Capillary tubes, micropipettes, or equivalent

15.3.4. UV light box (long and short wave)

15.3.5. TLC solvent systems - The following solvent systems are approved for use. Additional solvent systems may be used but the recipes must be noted in the case file.

15.3.5.1. T1 – Methanol/NH<sub>4</sub>OH (100:1.5) – general screening

15.3.5.2. T7 – Benzene/Dioxane/95% EtOH/NH<sub>4</sub>OH (10:8:1:1) – general screening

15.3.5.3. FM2 – Chloroform/Methanol (9:1) – general screening

15.3.5.4. TD – Chloroform/Acetone (4:1) – benzodiazepines

15.3.5.5. RW2 – Hexanes/Diethyl Ether (4:1) – cannabinoids



15.3.6. TLC indicator reagents - The following indicator reagents are approved for visualization. Additional indicator reagents may be used but the recipes must be noted in the case file.

15.3.6.1. Iodoplatinate reagent

15.3.6.1.1. Recipe: Dissolve 0.25 g chloroplatinic acid and 5 g potassium iodide in 100 ml water.

15.3.6.1.2. Developed spots appear purple.

15.3.6.2. Van Urk's Reagent (same as the reagent used for the chemical spot test)

15.3.6.2.1. Recipe: Dissolve 4 g p-dimethylaminobenzaldehyde in 450 ml 95% ethanol. Very slowly add 50 ml concentrated sulfuric acid.

15.3.6.2.2. Plate may be heated after spraying to increase the intensity of the spot. The color of the spot will be the same as for the chemical spot test.

15.3.6.3. Fast Blue Salt B Reagent (1%)

15.3.6.3.1. Recipe: Dissolve 1 g Fast blue B salt in 100 ml water.

15.3.6.3.2. Developed spot for THC appears red.

**15.4. Standards and Controls**

15.4.1. An appropriate known standard will be analyzed at the same time and under the same conditions as case samples to test the solvent systems and indicator reagents used. The appearance of the standard on a visualized TLC plate will indicate that the solvent systems and indicator reagents are working properly.

**15.5. Procedure**

15.5.1. In general, the following steps are taken when performing TLC on case samples:

15.5.1.1. Extract the sample with an appropriate solvent.

15.5.1.2. 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.

15.5.1.3. Allow the sample and standard to dry after application.

15.5.1.4. Place the plate vertically into a covered beaker/tray with enough liquid from the solvent system being used to cover 0.5 to 1.0 cm of the sample end of the plate.





15.5.1.5. Allow the solvent front to rise near the top of the TLC plate.

15.5.1.6. 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.

15.5.1.7. Spray the dried plate with an appropriate indicator reagent and/or view under UV light to visualize the component(s) of interest.

#### 15.5.2. Analysis and Interpretation

15.5.2.1. Examine the visualized TLC plate to locate sample and standard spots. If spots cannot be visualized, then the TLC may need to be re-run using more sample or standard, a different solvent system, a different medium, or a different indicator reagent. Spots that are visualized should be examined to ensure that they are acceptable for comparison and not smeared, streaked, or too concentrated.

15.5.2.2. Compare the location of the sample spot to that of the standard. A positive determination is made when a sample spot matches the color and location of the standard spot. If the sample and standard spots do not have the same color and location when compared or if the spots are not acceptable for comparison, then a positive determination cannot be made.

15.5.2.3. The results of analysis will be documented on the **TLC Sheet** which will include the following information:

**Case** – This is the unique case identifier.

**Date** – This is the date that observations are made. Any observations on a different date than this will be documented accordingly next to the corresponding run number.

**Analyst** – Placement of initials indicates the person(s) who made the documented observations.

**Item Number** – The LIMS generated item/sub-item number for the exhibit(s).

**Run Number** – Each spotting of a sample and corresponding standard will be given a run number. An item may have more than one run number due to multiple attempts or multiple sample/standard comparisons (e.g. one item of powder has both cocaine and heroin but three attempts are made for the heroin so there would be a total of 4 runs).

**Sample Preparation** – Documentation of how the sample was prepared such as solvent or extraction used (e.g. in ME OH, base extr into CH<sub>2</sub>Cl<sub>2</sub>, from GCMS vial).



**Plate Medium** – This is the type of TLC plate used (e.g. plastic, glass).

**Solvent System** – A mark in the FM2 or T7 box will indicate which solvent system was used. If another system is used it is noted under Other.

**Visualization** – A mark in the UV or Spray box will indicate how the spots were visualized (both may be checked). If Spray is marked, then the type of reagent used will also be indicated in the corresponding box.

**Standard Used # / Name** – The drug standard used for comparison to the sample is documented here by in-house identification number and name (e.g. 11B meth HCl).

**Observations** – Space is provided to document the results of comparison between the sample and standard spots. Additional space is provided to indicate reasons why spots may not be suitable for comparison.

**Will be Re-Run** – A box is provided to mark if a sample will be re-run.

### 15.6. Limitations

15.6.1. The results of TLC are not considered to be specific in nature and further structural confirmation by instrumental analysis is necessary for the positive identification of a questioned substance.

15.6.2. Various factors can affect the final results 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.

### 15.7. Advantages

15.7.1. TLC is a relatively quick and easy technique.

15.7.2. It can be used as a clean-up procedure for complex mixtures.

15.7.3. It requires no expensive instrumentation.

### 15.8. Literature and Supporting Documentation

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

15.8.2. A.C. Moffat, "Thin-Layer Chromatography" in Clarke's *Isolation and Identification of Drugs*, 2nd edition (London: the Pharmaceutical Press, 1986), 160-177.

15.8.3. Fox, R. H.; "Paper Chromatography", in *Isolation and Identification of Drugs*, ed. E.G.C. Clarke (London: The Pharmaceutical Press, 1969), 43-58.



- 15.8.4. Miller, J. A.; Neuzil, E. F., *Organic Chemistry, Concepts and Applications*, (D.C. Heath & Company, Lexington, Mass., 1979), 555.
- 15.8.5. "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.
- 15.8.6. "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.
- 15.8.7. E. Buel, C. N. Plum, and S. K. Frisbie, "An Evaluation of a Partition Thin Layer Chromatography System for the Identification of Cannabinoids", *Microgram*, 15 (1982): 145-157.
- 15.8.8. R.B. Hughes and R.R. Kessler, "Increased Safety and Specificity in the Thin-layer Chromatographic Identification of Marihuana", *Journal of Forensic Science*, 24 (1979): 842-846.
- 15.8.9. R.B. Hughes and V.J. Warner, Jr., "A Study of False Positives in the Chemical Identification of Marihuana", *Journal of Forensic Science*, 23 (1978): 304-310.

#### **15.9. Related Documents**

- 15.9.1. Reagent Logbook
- 15.9.2. Examination Sheet
- 15.9.3. TLC Sheet



## 16. Excess Quantity Cases

### 16.1. Scope

16.1.1. To provide guidelines for handling excess quantity cases.

### 16.2. Policy

16.2.1. An excess quantity case is defined as any case for which a representative sample must be taken and preserved. The evidence will be photographed, analyzed, and handled in accordance with established laboratory procedures and Texas Drug Laws, Health and Safety Code Section 481.160: Destruction of Excess Quantities.

Note: If a latent print examination is requested, the analyst should consult with the Latent Print section and the Seized Drugs section manager, section supervisors, or designee regarding the handling and transfer of evidence for processing.

### 16.3. Procedure

It is recommended that two analysts fully process a case identified as an excess quantity case, one will be the primary receiving analyst and the other will be a secondary assisting analyst. Processing of an excess quantity case by only one analyst will require the approval of the section manager, section supervisors, or designee and the approval will be documented in the case record.

16.3.1. The primary receiving analyst and the secondary analyst will place the unique case identifier and their initials on all exhibits.

16.3.2. The analysts will ensure that the case is photographed. The photograph should reasonably demonstrate the entire case. If all containers cannot be encompassed in one photograph, overlapping photographs should be taken. If the case is processed in parts due to space or time constraints, then each part should be photographed and documented separately to represent the whole. Digital photographs are acceptable as long as individual items can be distinguished. Photographs will be labeled to include the unique case identifier and item designators, analysts' initials, and the date the photos were taken (this information may be included within the photograph in lieu of labeling printed photographs). A videotape may be taken at any time at the discretion of the analyst.



16.3.3. Weights of all items will be observed and verified by both analysts. All bundles will be grouped according to size and appearance. A reasonable packaging tare weight will be determined for each bundle grouping.

16.3.3.1. To determine a reasonable tare weight:

The packaging from at least one of the largest packages in each bundle group will be completely removed and weighed. At this point, the bundle should be broken apart to check for consistency throughout the whole bundle. The decision whether or not to open other bundles completely due to apparent lightness, heaviness, or appearance will be at the discretion of the analyst.

16.3.4. If the total weight for the case is near one of the weights used as a cut-off in the Texas Health and Safety Code, the receiving analyst will determine the appropriate weighing method.

16.3.5. The sampling and analysis of all exhibits will be observed by both analysts. Refer to the Analysis Guidelines section for the appropriate sampling and analysis procedures depending on the type of evidence submitted (powder, plant substance, liquid, etc.).

16.3.6. After weighing and analysis of the evidence is completed, the representative samples will be assembled and preserved. Both analysts will observe and verify the collection and weighing of the representative sample and initial appropriately on the Seized Drugs section worksheets used.

16.3.6.1. **To determine an appropriate representative sample:**

16.3.6.1.1. The representative sample will consist of a minimum of five separate containers randomly sampled from the total amount of evidence.

16.3.6.1.2. If the contents of five total original containers meet the representative sample requirements outlined under **Retention of Samples**, these intact containers may be saved as the representative sample. If less than five intact containers are available to provide the sample required, the analyst makes up the difference for the representative sample with samplings from the remaining excess quantity controlled substance. Refer to **Retention of Samples** for requirements to prepare representative samples for specific types of controlled substances.



16.3.6.1.3. Evidence that consists of one single container of liquid will require the taking and preserving of only one representative sample.

16.3.6.1.4. Any items that are not bulk-wrapped (i.e., baggies, pipes, etc.) will be retained as part of the representative sample. An appropriate notation will be made for each item.

16.3.6.1.5. Part of the representative sample should be composed of an intact parcel of the excess quantity case, if possible (i.e., one brick, one bundle, etc.).

16.3.6.1.6. If a large excess quantity case is composed of evidence from multiple addresses, retain a representative sample from each source.

16.3.7. At least one set of initials from all submitting officers, if available, any initials documenting transfers of evidence, and the initials of the receiving analyst will be retained with the representative sample. The initials will be either examples of the initials cut from the original packaging or a photograph of the initials. The representative sample will be labeled as "**Representative Sample.**"

16.3.8. The remainder of the case will be packaged as **excess quantities** as follows:

16.3.8.1. The container size for excess quantities should be limited to forty pounds.

16.3.8.2. The following information should be on each container:

16.3.8.2.1. Analysts' initials and unique case identifier

16.3.8.2.2. Notations of "**1/5, 2/5, etc.**" or "**1 of 5, 2 of 5, etc.**" or **the original submitting agency identifiers** to identify multiple containers of the same case

16.3.8.2.3. Notation of "**Excess**" may also be included

16.3.8.2.4. The required information on the containers should be clearly visible. Use labels to place the required information on dark containers. All information on the plastic bags should be covered with tape. All bags should be deflated as much as possible.



#### **16.4. Retention of Samples**

16.4.1. The total amount to be retained as a representative sample will be determined by the submitting agency. The analyst should consult with the section manager, section supervisors, or designee to clarify any questions regarding how much sample to retain for a specific case. The following are general guidelines to use in the absence of other case specific requests.

##### **16.4.2. Excess Quantity Plant Substance:**

16.4.2.1. An amount to exceed 50 pounds should be retained as a representative sample. At least five separate containers must be present (Health and Safety Code Section 481.160).

16.4.2.2. Fresh plant substance will be dried, and all roots, dirt, and stalks removed prior to weighing (stalks are the large woody stems that test negative for THC). At least five separate containers must be saved.

16.4.2.3. In the case of some excess quantity plant substance cases such as Khat, it may be necessary to retain the representative sample in the freezer.

##### **16.4.3. Excess Quantity Powders:**

16.4.3.1. One intact kilogram package and 4 small bags should be retained as a representative sample. At least five separate containers must be present. If the excess quantity powder case does not contain kilogram packages, over 400 grams and at least 5 packages must be retained.

16.4.3.2. For powder cocaine identified for federal prosecution, eleven kilogram packages should be retained as a representative sample.

##### **16.4.4. Excess Quantity Liquids:**

16.4.4.1. At least 500 milliliters (at least 400 grams) should be retained as a representative sample (chemical precursors or liquid controlled substances).

16.4.4.2. If the excess quantity liquid is in only one container, only one sample of at least 500 milliliters (at least 400 grams) should be retained.



**16.4.5. Tablets and Capsules:**

16.4.5.1. At least 400 grams of any controlled substance tablet or capsule should be retained as a representative sample. At least five separate containers must be present. For large numbers of non-controlled substance tablets or capsules, usually a small representative sampling is sufficient.

**16.5. Reporting**

16.5.1. The report of analysis for an excess quantity case should follow the Reporting Guidelines section as usual.

**16.6. Return of Evidence to Submitting Agency**

16.6.1. The analyst will submit the representative sample and remaining excess portions to the original submitting agency for subsequent handling.





**17. Clandestine Laboratories (Rescinded as of August 16, 2004)**



## **18. Weighing Practices and Estimation of the Uncertainty of Measurement**

### **18.1. Scope**

18.1.1. To describe basic weighing practices as well as procedures for the determination of the estimation of the uncertainty of measurement.

### **18.2. Practices for Weighing Samples**

18.2.1. Select the appropriate balance for the amount of sample to be weighed. Analytical, top-loading, and bulky (high-capacity) balances are acceptable for routine casework.

18.2.2. Inspect the balance for cleanliness and ensure that the necessary checks have been performed.

18.2.3. The balance used will be recorded in the case notes.

18.2.4. Weights will be recorded in the case notes as they are displayed on the balance. Calculations involving weights will be done using the weights as they are recorded.

18.2.5. Weights will be noted as net weight (without packaging) or as gross weight (with packaging). Notations of the gross weight will refer to the substance(s) and the inner most container(s) unless otherwise specified.

18.2.6. Whenever feasible casework samples will be weighed using the static weighing process to reduce the likelihood of sample spillage onto the balance. The static weighing process follows these outlined steps:

18.2.6.1. The taring of a weighing vessel on the balance

18.2.6.2. Removal of the weighing vessel from the balance

18.2.6.3. Addition of sample to the weighing vessel

18.2.6.4. Return of the weighing vessel with sample to the balance

18.2.6.5. Recording of the weight reading

18.2.7. Under certain circumstances a dynamic weighing process may be more practical. The dynamic weighing process follows these outlined steps:

18.2.7.1. The taring of a weighing vessel on the balance

18.2.7.2. Addition of sample directly to the weighing vessel while it's still on the balance



18.2.7.3. Recording of the weight reading

18.2.8. For select samples as in Excess Quantity cases the direct addition of sample to the balance may be the preferred process for weighing.

18.2.9. It is not necessary to record the weighing process in the case notes.

18.2.10. In instances where statutory requirements designate weight thresholds (cut-off weights), sufficient samples will be weighed and analyzed to exceed the threshold.

18.2.10.1. Net weights for up to 10 samples within a grouping should be determined separately, if feasible.

18.2.10.2. For more than 10 samples within a grouping, the net weight may be calculated by use of a packaging tare weight.

18.2.11. If a packaging tare weight is used to determine a net weight from the gross weight, then the determination of the packaging tare weight will be documented in the case notes. One method of determining a packaging tare weight for multiple samples is as follows:

18.2.11.1. Choose three representative containers from the grouping

18.2.11.2. Record the weight of the three empty containers and determine the average

18.2.11.3. Multiply the number of samples in the grouping by the average to determine the total packaging weight.

18.2.11.4. The calculated total packaging weight can then be subtracted from the total gross weight of the grouping to determine the total net weight.

18.2.12. For Excess Quantity cases the weights and packaging tare weights will be determined as appropriate for each case and documented in the case file. Consult with the section manager, section supervisors, or designee for clarification if necessary.

18.2.13. Conversion factors to be used include the following:

18.2.13.1. 28.35 g = 1 oz

18.2.13.2. 1 kg = 2.2 lb

18.2.13.3. 453.6 g = 1 lb



### 18.3. Estimation of the Uncertainty of Measurement (UM)

18.3.1. For those substances that have weight thresholds as listed in the Texas Health and Safety Code Section 481 an estimation of the UM is determined for their net weights.

18.3.2. The estimation of the UM for weight determinations will be evaluated at least annually or when a new balance is placed into service using the following guidelines:

18.3.2.1. In-house studies will be performed to document contributions to the UM from both random (Type A) and systematic (Type B) sources for each balance. These values will be used to determine a combined uncertainty using the root sum square method.

18.3.2.2. To determine the expanded uncertainty, the combined uncertainty will be multiplied by a coverage factor ( $k = 2$ ) for a confidence level of 95.45%.

18.3.2.3. The use of the static weighing process is included in the determination of the UM by multiplying the expanding uncertainty for each balance type by an additional factor of 2. In those instances where the dynamic weighing process or direct placement of sample onto a balance is used, this factor will be retained and will result in an overestimation of the UM.

18.3.2.4. The static expanded uncertainty values will be determined by using the **Balance Uncertainty Budget Form** for each type of balance.

18.3.3. The value to be used for the estimation of UM will be determined for each type of balance and will be based on the largest value obtained from the historic studies.

18.3.4. To determine the final total expanded uncertainty for a weight measurement, the static expanded uncertainty for the balance type used will be multiplied by the number of weighing events.

18.3.5. Single Sample

Since weighing one sample consists of one weighing event, the calculated total expanded uncertainty will be the static expanded uncertainty for the type of balance used times one.



#### 18.3.6. Multiple Samples

When weights are combined to determine a total net weight, their individual associated uncertainty values will be taken into account to determine the total expanded uncertainty. The static expanded uncertainty for the type of balance used will be multiplied by the total number of weighing events to calculate the total expanded uncertainty.

18.3.7. The units for the estimation of the UM will be determined in the same units as the recorded weight.

18.3.8. The total expanded uncertainty will be determined to the same number of decimal places as the readability of the balance used.

18.3.9. Calculations and final values for the estimation of the UM will be included in the case file.

18.3.10. If the total expanded uncertainty is equal to or larger than the weight, a more accurate balance will be used or the sample will be reported as “trace” as appropriate.

18.3.11. The values for the estimation of the UM will be included on the final report if the upper or lower limit value could result in a weight that is in a different penalty range than the reported net weight. A statement of the level of confidence such as “Measurement uncertainty of weight measurements are reported at a 95.45% level of confidence” will also be included on the report in these cases.

#### **18.4. Literature and Supporting Documentation**

18.4.1. SWGDRUG, *Measurement Uncertainty for Weight Determinations in Seized Drug Analysis Supplemental Document SD-3*, Revision 2, 2011-07-07.

#### **18.5. Related Documents**

18.5.1. Balance Uncertainty Budget Form



## 19. Reporting Guidelines

### 19.1. Scope

19.1.1. To establish standards for reporting the results from the analysis of controlled substances, dangerous drugs, botanical material, and other chemical substances examined by analysts in the Seized Drugs section.

### 19.2. Procedure

19.2.1. Reports of analysis are entered into the Laboratory Information Management System (LIMS).

19.2.2. The exhibits related to a case will be identified on the report by their assigned Item designators, quantity, and description whether analyzed or not.

19.2.3. Under **Results and Interpretations** all appropriate results will be entered.

19.2.4. The name, title, and signature of the analyst will be noted at the end of the report.

### 19.3. Reporting Guidelines for Analytical Results

19.3.1. Reporting guidelines for controlled substances are based on the statutes and definitions provided in the *Schedules of Controlled Substances* which are maintained by the Texas Department of State Health Services and in Chapters 481-485 of the *Texas Health and Safety Code (HSC)* which contains the *Texas Controlled Substances Act*. The statutes determine the terminology used in reporting the identification of most controlled substances and requires the net weight of that substance to establish the penalty group.

#### 19.3.2. General Reporting Examples of Identification

19.3.2.1. Report the identification of a controlled substance as it appears in the *Texas Controlled Substances Act* or the *Schedules of Controlled Substances*. If there is a question about how to report a substance or there is a difference in how a substance is listed in the statutes consult with the section manager, section supervisors, or designee.

19.3.2.2. Precede the name of all substances identified with the word "**Contains**". Cannabis sativa L., **marihuana**, and peyote will not be preceded with "contains" unless they contain other materials.



19.3.2.3. If more than one controlled substance is identified in a sample, report them all after **"Contains"**.

*Examples: Contains amphetamine and methamphetamine  
Contains cocaine and phencyclidine  
Contains cocaine and **marihuana***

19.3.2.4. If a controlled substance and a dangerous drug are identified in a sample, the analyst should normally report only the controlled substance and note the presence of the dangerous drug on the **Examination Sheet**. At the discretion of the analyst, it may be necessary to report other substances identified for certain cases.

19.3.2.5. 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, and include the notation that they are dangerous drugs.

*Example (for Viagra): Contains sildenafil – Dangerous Drug*

### 19.3.3. Reporting Cannabis sativa L. and **Marihuana**

19.3.3.1. Report plant substance identified as Cannabis sativa L. as "Cannabis sativa L." (not **"contains Cannabis sativa L."**). **Report plant substance identified as marihuana as "Marihuana" (not "contains Marihuana").** Report the net weight in metric units and ounces or pounds.

19.3.3.2. If a significant amount of an impurity, such as tobacco, is present in the Cannabis sativa L./**marihuana** sample (and cannot be readily separated), make a conservative visual or microscopic estimate of the percent of Cannabis sativa L. /**marihuana** present, note this on the **Examination Sheet**, and report the total net weight in metric units and ounces or pounds. Report the substance beginning with the word **"Contains"** and add an appropriate footnote:

*Example: Contains Cannabis sativa L. \**

*\*Visually estimated to be 33% of the reported weight*



#### 19.3.4. Reporting Peyote Samples

19.3.4.1. 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"** along with the weight in grams.

#### 19.3.5. Reporting Mushroom Samples

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

#### 19.3.6. Reporting Opium Samples

19.3.6.1. 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. Samples which contain heroin should be reported as **"Contains heroin"**.

#### 19.3.7. Reporting Derivatives of Barbituric Acid

19.3.7.1. There are a number of derivatives of barbituric acid that are listed by name in the *Texas Controlled Substances Act*. In those cases, report the name of the barbiturate identified (for example, **"Contains secobarbital"**). If the barbiturate is not listed by name, such as butalbital, then it should be reported as **"Contains butalbital - a derivative of barbituric acid"**.

#### 19.3.8. Reporting Derivatives of 2-aminopropanal

19.3.8.1. Report the name of the compound identified with the notation that it is a derivative of 2-aminopropanal. The isomer form does not need to be identified as all isomers are derivatives.

*Example: Contains methylenedioxy-N-methylcathinone (a derivative of 2-aminopropanal)*

*Contains methylethcathinone (a derivative of 2-aminopropanal)*





#### 19.4. Reporting Weights

- 19.4.1. If a controlled substance or dangerous drug is identified in a sample, then report the net weight of the sample.
- 19.4.2. If tablets and capsules are identified, then include the net weight on the report. The number of tablets and capsules as well as the number of containers should also be reported. It is acceptable to describe the number of tablets and capsules as numerous when the number is too large to count (see Tablets and Capsules – General in the Analysis Guidelines section for additional information).
- 19.4.3. For most substances report the net weight in grams if it is less than 1,000 grams. Weights greater than or equal to 1,000 grams may be reported in grams or in kilograms. Residue amounts should be reported as **trace**.
- 19.4.4. For Cannabis sativa L./**marihuana** and Penalty Group 2-A substances (synthetic cannabinoids) weights that are determined in metric units will be converted to ounces or pounds and both units will be included on the report. For samples weighing less than one pound, report the weight in ounces. Samples weighing more than one pound are typically reported in pounds to at least one decimal place instead of in ounces. If a sample weighs less than 0.01 ounces, the analyst will report the weight as "**Less than 0.01 ounces**".

*For example:*

<i>Bag with plant substance</i>	<i>3.52 grams / 0.12 ounces</i>	<i>Contains JWH-018</i>
<i>Bundle with plant substance</i>	<i>3.316 kilograms / 7.2 pounds</i>	<i><b>Marihuana</b></i>
<i>Cigar</i>	<i>0.21 grams / Less than 0.01 ounces</i>	<i>Cannabis sativa L.</i>

#### 19.5. Reporting Abuse Units

- 19.5.1. Report the number of abuse units for substances identified as belonging to Penalty Group 1-A 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 a liquid, 40 micrograms is one abuse unit.

#### 19.6. Miscellaneous

- 19.6.1. Dilutants (diluent) 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.



19.6.2. 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.

19.6.3. For certain substances, it is necessary to know the isomer form present to establish the appropriate penalty group or identification (e.g. dextropropoxyphene, dextromethorphan, citalopram, and escitalopram). If pharmaceutical information is used to determine the isomer form present, then the report should include an appropriate footnote, such as:

*"Isomer identified by pharmaceutical information"*

19.6.4. In tablets, capsules and liquid pharmaceutical preparations containing a controlled substance, it is sometimes necessary to know the amount of the controlled substance present to establish the penalty group as stated in the *Texas Controlled Substances Act*. The amount present may be determined by accepted analytical quantitation procedures or by available pharmaceutical information.

If pharmaceutical information is used (quantitation not performed), an appropriate footnote should be included in the report, such as:

*"Pharmaceutical identification indicates not more than 200 milligrams of codeine per 100 milliliters or 100 grams and includes one or more nonnarcotic active medicinal ingredients."*      or

*"Pharmaceutical identification indicates 800 milligrams per dosage unit."*

When pharmaceutical information is not available (as in the case of a crushed tablet) and quantitation is not performed, then report the substances identified in the exhibit after "Contains". An appropriate footnote may be added.

*Example: Contains codeine and promethazine  
                  Contains dihydrocodeinone and acetaminophen*

19.6.5. Steroids and steroid esters may be reported by the steroid alcohol name or by the identified steroid ester.



*Example: Contains testosterone or Contains testosterone enanthate  
Contains nandrolone or Contains nandrolone decanoate*

19.6.6. If a sample is examined for the presence of an **abusable volatile chemical** as listed in HSC 485, and one is identified, then report the results of the substance identified with the notation that it is an abusable volatile chemical. No weight is necessary on the report.

*Example: Contains toluene – An Abusable Volatile Chemical*

19.6.7. Items for which visual examination by two analysts indicates that no sample / residue is present for analysis should be reported as **“No analysis performed (no visible sample).”**

19.6.8. When field testers are received without any other evidence to analyze, they should be reported as **“No unprocessed sample available for analysis.”**

19.6.9. Items for which visual examination by two analysts indicates that plant substance has undergone excessive decomposition should be reported as **“No analysis performed due to excessive decomposition”**.

19.6.10. Exhibits that are not analyzed are reported as **“Retained with no analysis”** and no weights need to be reported. Alternatively, the following statement may be added to the report:

*“Items of evidence not listed under Results and Interpretations were not analyzed.”*

19.6.11. Samples may be reported as **“No controlled substance identified”** after the sample has been subjected to sufficient analytical examinations. No weights need to be reported and an appropriate footnote may be added at the discretion of the analyst, for example:

*“Analysis indicates the presence of the following non-controlled substance(s): benzocaine and caffeine”*

19.6.12. If a substance has been subjected to pharmaceutical identification without structural confirmation, the report will reflect **“Indication [substance]”**. If a dangerous drug or over the counter substance is indicated, then the report will include the notation that the substance is a dangerous drug or an over the counter product. A notation will be added to indicate that only presumptive testing was performed.



*Example: Indication amitriptyline – Dangerous drug*  
*Indication acetaminophen – Over the counter*

19.6.13. In the situation where a structural test is unavailable by the laboratory to support pharmaceutical identifications (insulin, human growth hormone, new products without published characterizations), the report should include the available information with an appropriate footnote:

*Example: Indication levothyroxine – Dangerous drug \**

*\* Pharmaceutical identification only. Complete analysis is not possible by this laboratory.*

19.6.14. Items for which visual examination by two analysts indicates that insufficient sample / residue is present for analysis and for retesting should be reported as **“No analysis performed (Insufficient sample for analysis and retesting).”**

19.6.15. **Plant substance items suspected of being Cannabis sativa L./marihuana that have a net weight less than 0.20 grams (or trace for a residue) should be reported as “No analysis performed (Insufficient sample for analysis and retesting).”**

19.6.16. Documentation is to be included on the report to reflect the analytical scheme (methods) and sampling plan used as appropriate.

## 19.7. Footnotes

19.7.1. The following is a list of certain footnotes that will be available for inclusion on the report:

19.7.1.1. *Pharmaceutical identification indicates: Not more than 1.8 grams of codeine, or any of its salts, per 100 milliliters or not more than 90 milligrams per dosage unit, with one or more active, nonnarcotic ingredients in recognized therapeutic amounts.*

19.7.1.2. *Pharmaceutical identification indicates: Not more than 300 milligrams of dihydrocodeinone, or any of its salts, per 100 milliliters or not more than 15 milligrams per dosage unit, with one or more active, nonnarcotic ingredients in recognized therapeutic amounts.*

19.7.1.3. *Not more than 200 milligrams of codeine per 100 milliliters or 100 grams and includes one or more nonnarcotic active medicinal ingredients.*



19.7.1.4. *Reported results are based on presumptive testing only. If further analysis is required, please contact this laboratory as soon as possible.*

19.7.1.5. *Weight includes paper.*

19.7.1.6. *An analogue of gamma-Hydroxybutyric Acid (gamma-Hydroxybutyrate).*

19.7.1.7. Specialized footnotes may be used with the approval of the section manager, section supervisors, or designee.



20. Abbreviations

20.1. Scope

20.1.1. To provide a list of useful abbreviations.

20.2. General Abbreviations

~	Approximately
AB	Analytical Balance
A/B extr	Acid/Base extraction
ACLS	Amera-Chem Library Search
approx	Approximately
AR	Administrative review
au	abuse unit(s)
AVC	Abusable Volatile Chemical
BB	Bulky Balance
crt/ct	Court
DD	Dangerous Drug
DIB	Drug Identification Bible
disp	Disposed
dism	Dismissed
EMS	Evidence Management System
est	Estimate(d)
ETAC	Ethyl acetate
EXI	Extraneous ions
Extr	Extracted or Extraction
FAD	Forensic Analysis Division
FID	Flame Ionization Detector
FCN	Forensic case number
FTIR	Fourier Transform Infrared (Spectrophotometry)
g	Grams
GC	Gas chromatograph
gr	Gross/Gross Weight
gross	Gross weight
HFSC	Houston Forensic Science Center
Ind	Indication
INI	Insufficient ions
Inv	Investigation
ISTD	Internal Standard
JIMS	Justice Information Management System



juv .....	juvenile
kg .....	Kilograms
lb(s) .....	Pounds
L .....	Liters
LIMS .....	Laboratory Information Management System
mg .....	Milligrams
ml .....	Milliliters
MS .....	Mass spectrometer
MT .....	Mettler Toledo top-loading balance
NAM .....	No acceptable match or Not an acceptable match
NAP .....	No Analysis Performed
NCS .....	No Controlled Substance
NCSI .....	No Controlled Substance Identified
net .....	Net/Net weight
NVS .....	No Visible Sample
neg .....	Negative
oz/ozs .....	Ounces
PDR .....	Physician's Desk Reference
PHI .....	Pharmaceutical Identification
pos .....	Positive
RT .....	Retention time
Rx .....	Prescription
STD .....	Standard
TB .....	Top-Loading Balance
TIC .....	Total Ion Chromatogram
TLC .....	Thin layer chromatography
TR .....	Technical review
UM .....	Uncertainty of Measurement
UV/VIS .....	Ultraviolet/Visible (Spectrophotometry)
wh .....	White
wt .....	Weight

### **20.3. Abbreviations for Evidence Documentation**

bot(s) .....	Bottle(s)
c (with line above) .....	Containing and/or with
cap(s) .....	Capsule(s)
cb(s) .....	Cardboard box
ch .....	Chunk
cig(s) .....	Cigarette(s)



cig stub(s) .....	Cigarette Stub(s)
cry .....	Crystalline
ee .....	evidence envelope
env .....	envelope
evi .....	evidence
liq .....	Liquid
num .....	numerous
pkg .....	Package/Packing/Packaging
pl(s) .....	Plastic(s)
PS .....	Plant Substance
s .....	Sealed
sub .....	Substance
tab(s) .....	tablet(s)
zip(s) .....	Ziploc(k)(s)

#### 20.4. Abbreviations for Drugs

(This is not intended to be an exhaustive list as many substances have commonly accepted or otherwise documented abbreviations)

1,4-BD .....	1,4-butanediol
2 C-B .....	4-bromo-2,5-dimethoxyphenethylamine
2 C-E .....	4-ethyl-2,5-dimethoxyphenethylamine
2 C-I .....	4-iodo-2,5-dimethoxyphenethylamine
acet .....	Acetaminophen
alp/alpz .....	alprazolam
amp/amph .....	Amphetamine
APAP .....	Acetaminophen/acetyl-para-aminophenol
BZP .....	Benzylpiperazine
coc .....	Cocaine
cod .....	Codeine
CPP .....	Chlorophenylpiperazine
CSL .....	Cannabis sativa L.
DBZP .....	1,4-Dibenzylpiperazine
dhy .....	Dihydrocodeinone
DMS .....	Dimethylsulfone
GBL .....	gamma-butyrolactone
GHB .....	gamma-hydroxybutyric acid( $\gamma$ -hydroxybutyrate)
LSD .....	Lysergic Acid Diethylamide
mari/marih .....	Marihuana





MDA.....	3,4-Methylenedioxy amphetamine
MDMA .....	3,4-Methylenedioxy methamphetamine
MDE .....	3,4-Methylenedioxy N-ethylamphetamine
MDP2POL.....	3,4-Methylenedioxy phenyl-2-propanol
MeOPP .....	Methoxyphenylpiperazine
meth .....	Methamphetamine
PCP.....	Phencyclidine
prom/prometh .....	Promethazine
syn cann.....	Synthetic cannabinoid
TFMPP.....	1-(3-Trifluoromethylphenyl)piperazine
THC .....	Tetrahydrocannabinol



**21. Counting of Items and Tests (Rescinded as of October 20, 2014)**



## 22. Re-analysis of Cases

### 22.1. Scope

22.1.1. To provide guidelines for conducting re-analysis of cases under various circumstances.

### 22.2. Re-analysis for Purposes of Testifying in Court

22.2.1. The following guideline is provided to aid in the re-analysis of cases when the original analyst is not available to testify in court.

22.2.1.1. The Seized Drugs section manager, section supervisors, or designee will assign the case to an analyst for testing.

22.2.1.2. The new analyst will process the case following normal procedures for analysis and documentation.

22.2.1.3. The new analyst will report findings in a new report as usual with the addition of a statement at the beginning of the report to explain the reason for the re-analysis. The following wording may be used as an example:

*“On (date), Title (name), PR# was requested by ADA John Doe to re-analyze evidence in this offense for the purpose of testifying in an upcoming trial. The Laboratory no longer employs the original analyst.”*

### 22.3. Re-analysis for On-going Quality Review or Investigation

22.3.1. The following guideline is provided to aid in the re-examination and re-analysis of cases conducted as a result of a quality review and/or investigation.

22.3.1.1. The evidence will be received from the appropriate personnel as directed by the Seized Drugs section manager, section supervisors, or Quality Division.

22.3.1.2. The evidence packaging with seals and the contents may be photographed, if directed or appropriate.

22.3.1.3. The assigned analyst will proceed with re-analysis of items as directed. Generally, the work previously conducted will be duplicated as much as possible following normal procedures for analysis and documentation.



22.3.1.4. The analyst will document the results as directed and will include a **Reanalysis Form**.

**22.4. Related Documents**

22.4.1. Reanalysis Form



**23. Guidelines for Processing Non-Active Cases (Rescinded as of December 1, 2016)**



## 24. Modification Summary

ISSUE DATE	CHANGE
Current Version	<p>Modifications to this version include but are not limited to the following changes:</p> <p>3.4.1 added “(see sections 3.8 – 3.10 for additional information about sampling of tablets and capsules)”</p> <p>3.4.6.5 added “Documentation that statistical sampling was used <u>including confidence levels and corresponding inferences regarding the population</u> is to be noted on the report.”</p> <p>3.7 updated to address analysis of marihuana and Cannabis sativa L.</p> <p>3.8.2 added “Tablets and capsules can typically be grouped based upon their appearance (size, color, and markings) <u>and/or packaging.</u>”</p> <p>6.6.1 added use of <b>Ideal Standard Tune</b> documents</p> <p>6.6.2 clarified that a standard check mix is run each day that samples are loaded</p> <p>6.7.6.1 clarified checks for analytical balances when infrequently used.</p> <p>6.7.6.2 clarified checks for top loading balances when infrequently used.</p> <p>6.8 pipettes section added</p> <p>6.10 added <b>Ideal Standard Tune</b> documents</p> <p>GC/MS moved from Section 8 to Section 7</p> <p>7.4 clarified tuning and standard check requirements</p> <p>7.9 added <b>Ideal Standard Tune</b> documents</p> <p>Added Section 8 GC/MS Decision-Point Assay for delta-9-THC in Plant Substance</p> <p>13.4.2 Koppanyi removed as frequently used reagent</p> <p>13.21.1 added “Quality-test with a known Cannabis sativa L. <u>or marihuana</u> sample.”</p> <p>Section 19 update reporting of marihuana</p> <p>19.3.3.3 deleted reporting of charred remains or trace amounts of Cannabis sativa L.</p> <p>19.3.3.4 deleted reporting of Cannabis sativa L. seeds</p> <p>20.2 Abbreviation for “ETAC” added for Ethyl acetate</p>



09-09-19	<p>Modifications to this version include but are not limited to the following changes:</p> <p>3.4.6.1 and 3.4.6.2 updated to include a discussion of random sampling</p> <p>3.11.4 Added the requirement for an additional GC/MS sample run when the initial GC/MS run shows the presence of acetaminophen without a controlled substance.</p> <p>13.12.2.3 updated to discuss the documentation of color reactions that occur with the addition of both reagents in the Marquis test</p> <p>13.12.3.10 add "...concentrated acid <u>that persists with the addition of the formaldehyde solution</u> indicates..."</p>
06-21-19	<p>Modifications to this version include but are not limited to the following changes:</p> <p>Changing "marihuana" to "Cannabis sativa L." throughout the document in response to HB 1325</p> <p><b>Marihuana Checklist</b> changed to <b>Cannabis sativa L. Checklist</b></p> <p>3.5.3 add use of separate portions for testing when possible</p> <p>3.7 add introductory discussion on the effect of HB 1325 on analysis of plant substance samples</p> <p>3.7.2 add "Generally, sample portions used for microscopic examination are also used for additional testing and this practice is not documented in the case notes."</p> <p>3.7.5 delete "...identification of THC in hashish samples,"</p> <p>11.2.2 This requirement includes plant substance samples <del>such as marihuana.</del></p> <p>13.21 updated for testing of material in general and not just plant material</p> <p>19.3.1 include reference to the <i>Schedules of Controlled Substances</i></p> <p>19.3.2.1 include reference to the <i>Schedules of Controlled Substances</i> and added "If there is a question about how to report a substance or there is a difference in how a substance is listed in the statutes consult with the section manager, section supervisors, or designee."</p> <p>19.3.3 delete "and Hashish"</p> <p>19.3.3.5 deleted reporting of hashish and liquid extracts</p> <p>19.4.4 and 19.4.5 combined</p> <p>20.4 Abbreviation for "CSL" added for Cannabis sativa L.</p>

End of Document