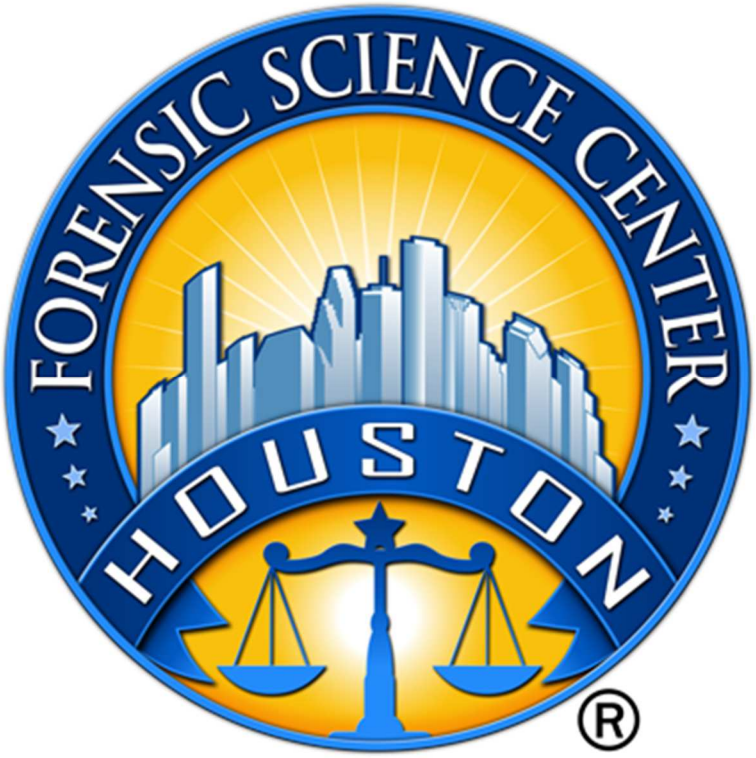


# Controlled Substances
# Standard Operating Procedures

Forensic Analysis Division

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## 1. Goals and Objectives

### 1.1. Goals

1.1.1. The primary goal of the Controlled Substances Section 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 controlled substance 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 controlled substance 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 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 completed case file review.

1.2.1.10. The time between receipt of evidence by an analyst and its preparation for return to the submitting agency should be less than one month. If evidence remains in an analyst's custody longer than one month, documentation should be included in the case file which explains the reason (for example, the evidence was being processed by latent prints either in part or in whole).

## 2. Evidence Handling

### 2.1 Scope

2.1.1. To provide guidelines for the handling of evidence in the Controlled Substances Section.

### 2.2. Receiving 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 a controlled substance 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. 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 controlled substance 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 **Controlled Substances 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 **Controlled Substances Inventory Sheet**.

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. 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.

2.2.2.9. 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.

2.2.2.10. 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 controlled substances 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. To prepare evidence for transfer to the Latent Print section, the analyst will separate packaging from the materials (powder, plant substance, etc.). If desired by the analyst, a Latent Print identification officer will be available to coordinate the separation and collection of item packaging in the presence of the analyst. 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, 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 **Controlled Substances 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. Controlled Substances Inventory Sheet

2.7.2. Controlled Substance 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 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.

3.4.2. The Controlled Substances 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. Use the above table to determine the number of randomly selected items for testing. Record the total net weight of the population and the total net weight of the randomly selected items and record on the **Controlled Substances Examination Sheet**.

3.4.6.2. Each randomly selected item is to be analyzed separately and completely.

3.4.6.3. 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.

3.4.6.4. Documentation that statistical sampling was used 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. See sections 3.8 – 3.10 for additional information about sampling of tablets and capsules.

3.4.9. 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. Data required for instrumental analyses

3.5.3.1. 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.

3.5.3.2. 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:

3.5.3.2.1. UV
All appropriate information regarding sample preparation, wavelengths, and absorbances will be documented on the UV printout or in the case file.

3.5.3.2.2. 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.

3.5.3.2.3. FTIR
All appropriate information regarding sample preparation and library/ literature comparisons will be documented on the FTIR printouts or in the case file.

3.5.3.2.4. 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. Documentation of internal standard runs will be maintained with the case file. Blanks run prior to the internal standard and sample runs will be maintained with the case file.

3.5.4. 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:

3.5.4.1. Thin Layer Chromatography

Each solvent system used is listed on the **Controlled Substances Examination Sheet**. The observations are documented as well as the standards used for comparison.

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

3.5.5. 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 **Controlled Substances Examination Sheet**.

3.5.6. 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 **Controlled Substances 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 **Controlled Substances 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 **Controlled Substances Examination Sheet**.

**3.7. Plant Substance and Plant Substance Residues**

3.7.1. Plant substance samples received for testing may be identified as marihuana 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 techniques to make a determination as to whether plant substance samples are or if they contain a controlled substance.

3.7.2. For the identification of marihuana, excluding seeds, microscopic identification and at least one other positive test (including the Duquenois-Levine chemical spot test or GC/MS) are required. Any features observed during microscopic examination of samples will be documented on the **Controlled Substances Marihuana Checklist**. For a microscopic examination to be positive a minimum of 2 physical characteristics must be observed including cystolithic hairs or glandular hairs.

3.7.3. Live plants:

3.7.3.1. Plants are dried before weighing and analyzing.

3.7.3.2. 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.

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

3.7.4. Germination of Marihuana Seeds

Seeds are identified by color, size, shape and surface pattern. The viability of seeds may be determined by germinating a sample of the seeds. If any seeds germinate, it is determined that the seeds are capable of beginning germination.

3.7.4.1. Select seeds for germination.

3.7.4.2. Place seeds between moistened filter paper or the equivalent, and place in an appropriate container.

3.7.4.3. Incubate at room temperature for up to 10 days.

3.7.4.4. Document the number of seeds that germinated.

3.7.5. Instrumental and non-instrumental methods may be used when necessary as in the identification of THC in hashish samples, plant substance suspected of containing synthetic cannabinoids, or residues which cannot be identified as marihuana using microscopic examination (see the basic analytical scheme noted in section 3.5).

3.7.6. 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.

3.7.7. 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 **Controlled Substances 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 marihuana weights are determined in metric units, they will be converted to ounces or pounds for the report.

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 **Controlled Substances 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). 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 **Controlled Substances 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 **Controlled Substances 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 **Controlled Substances 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 **Controlled Substances 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, 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.

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

**3.13. Related Documents**

3.13.1. Controlled Substances Examination Sheet

3.13.2. Controlled Substances Marihuana Checklist

## 4. Case Documentation

### 4.1. Scope

4.1.1. These policies are established as minimum requirements for case documentation and record keeping required for controlled substance 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. 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 **Controlled Substances 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 **Controlled Substances Examination Sheet**. Check for the presence of any necessary blanks.

4.3.1.5. All **Controlled Substances Examination Sheet(s)** and spectra must have the analyst's handwritten initials.

4.3.1.6. Verify that all observations listed on the **Controlled Substances 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 LIMS and/or in the case record.

4.3.3. Before giving any verbal results to a requestor (for example, priority, rush, or investigation cases) the analysis will be technically reviewed and the technical review will be documented as part of the case record. The results of analysis still need to be included in the test report.

**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. Unless approved in advance by the Forensic Analysis Division Director or designee, the technical and administrative review will not be conducted by the same individual. 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 **Controlled Substances 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 **Controlled Substances Examination Sheet** should be included in the report.

4.4.1.4. Verify that the correct information is listed for the inventoried evidence.

4.4.2. The completed administrative review is documented in the LIMS and/or in the case record.

**4.5. Administrative and Technical Review Issues**

4.5.1. 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, Supervisors, or designee, in person, 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 **Controlled Substances 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 **Controlled Substances Inventory Sheet(s), Controlled Substances Examination Sheet(s), Controlled Substances Notes Sheet(s),** instrument printouts, photographs, and other documents produced and used to reach a conclusion.

**4.8. Related Documents**

4.8.1. Controlled Substances Examination Sheet

4.8.2. Controlled Substances Inventory Sheet

4.8.3. Controlled Substances Notes Sheet

4.8.4. Controlled Substances Marihuana Checklist

## 5. Controlled Substances Worksheets

### 5.1. Scope

5.1.1. To provide guidelines for documentation of tests and observations on the **Controlled Substances Examination Sheet**, the **Controlled Substances Notes Sheet**, and the **Controlled Substances Marihuana Checklist**.

### 5.2. Controlled Substances 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 **Controlled Substances 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 marihuana (including cystolithic and/or glandular hairs) were observed. Neg indicates that insufficient or no characteristics for marihuana were observed. The characteristics observed as well as the

number of samples tested will be documented on the **Controlled Substances Marihuana 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 quantitation value 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** - The solvent system used to run thin layer chromatography plates is documented. Circle UV and/or Spray to note the visualization technique used. Circling Pos will indicate that the $R_f$ for the sample spot was the same as the $R_f$ for the standard spot. Circling Neg will indicate that the sample spot $R_f$ was not the same as the standard spot $R_f$. The standard name and number will be documented.

**# Sampled / Net Weight** – When a sampling plan is used and not all of the items within the group are sampled, then the actual number of items 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 **Controlled Substance 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 **Controlled Substance Examination Sheet** may be used following the proper guidelines for notations outlined above.

### 5.3. Controlled Substances 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 **Controlled Substances 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 **Controlled Substances 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. Controlled Substances Marihuana 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. Controlled Substances Examination Sheet

5.5.2. Controlled Substances Inventory Sheet

5.5.3. Controlled Substances Notes Sheet

5.5.4. Controlled Substances Marihuana Checklist

## 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)**

6.5.1. A performance verification check will be done daily for any operational GC instrument when in use. This may be performed through the use of check standards of known concentration. The mean concentration of these check standards will be calculated from three injections and the % relative standard deviation must be equal to or less than 10%. In addition the % difference of the mean from the known concentration must be equal to or less than 10%.

6.5.2. 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.

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

6.5.4. Run a solvent blank before all other runs and maintain a copy of the blank run with the case file.

6.5.5. Perform regular and preventive maintenance according to the manufacturer's recommendations. Records documenting all maintenance (e.g. column replacement and any major repairs) will be maintained in the section.

**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) according to established criteria for a successful tune. It is recommended that specifications used to check the instrument performance be kept next to the instrument for easy reference.

6.6.2. A standard check mix will be run daily when in use 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 the standard re-run.

6.6.3. Printed copies of tune records and daily 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.

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

6.7.6.3. The bulky scales (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 Scales 2,3 | 2 kg | 2.00 kg | ±0.02 kg |
| Bulky Scale 4 | 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.

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%RSD = 100 * (standard deviation / 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 at least annually.

6.8.4. Following an external calibration, or when a pipette/dispensette is being put back into service, or is being put into service for the first time an internal performance check must be conducted. The **Pipette/Dispensette Performance Check Worksheet** will be used for this purpose.

6.8.4.1. For fixed pipettes/dispensettes, 5 aliquot measurements of water at the specified fixed volume will be weighed on an analytical balance and recorded.

6.8.4.2. For adjustable pipettes/dispensettes, 5 aliquot measurements of water at 2 different volumes will be weighed on an analytical balance and recorded.

6.8.4.3. The % relative standard deviation (%RSD) is calculated for the recorded aliquot weights.

%RSD = 100 * (standard deviation / mean)

6.8.4.4. The acceptance criteria for the internal performance check will be the same as the value used by the external vendor to determine both an acceptable range for all aliquot weights and for the %RSD.

6.8.4.5. If a result from the check does not meet the acceptance criteria, first ensure that the pipette/dispensette is adjusted to the specified volume.

6.8.4.6. Check that the analytical balance used is level and clean and if applicable, use the internal calibration function of the balance prior to rechecking.

6.8.4.7. In addition, if the check does not meet the acceptable criteria, then the temperature conversion for the density of water may be considered.

6.8.4.8. If a result is outside of the acceptable range after performing the actions above, then the pipette/dispensette shall be immediately taken out of service until maintenance and/or calibration are performed by an external vendor.

6.8.5. Records documenting the results of the internal performance checks, maintenance and calibrations will be maintained in the section.

**6.9. Malfunction of an Instrument, Pipet, 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. Pipette/Dispensette Performance Check Worksheet

6.10.4. Equipment Service Form

## 7. Gas Chromatography (GC) for Quantitation of Codeine in Liquids

### 7.1. Scope

7.1.1. To establish a procedure to determine the concentration of codeine in liquid samples using an internal standard and gas chromatography/flame ionization detection (GC/FID).

### 7.2. Safety

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

7.2.2. Properly secure high-pressure gas cylinders.

7.2.3. Use caution around hot surfaces.

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 equipped with a flame ionization detector

7.3.2. Auto-sampler vials and caps

7.3.3. Injection syringe

7.3.4. Analytical balance needed for quantitation

7.3.5. Pipettes and Dispenser

7.3.6. Suitable solvents for sample preparation

7.3.7. Codeine drug standard

7.3.8. Octacosane (C28) hydrocarbon to be used as internal standard

### 7.4. Standards, Controls, and Calibration

7.4.1. A valid five point linearity plot using codeine base standards mixed with internal standard will be determined for the instrument. If major instrument repairs (e.g. replacement of the column or detector) are performed or if a fresh internal standard solution is prepared, the linearity will be re-confirmed.

7.4.2. Internal Standard Solutions

7.4.2.1. Internal standard stock solutions will be prepared by dissolving the C28 hydrocarbon in dichloromethane. The final concentration of the internal standard should be approximately 0.1 mg per mL of dichloromethane. If a fresh internal standard is prepared, then all standards and samples must be prepared using the new solution.

7.4.2.2. If the internal standard stock solution is stored for later use, it should be well sealed and not exposed to extreme temperatures. It will be labeled with the name of the internal standard, date of preparation, initials of the analyst who prepared the solution and the final concentration. The preparation of internal standard solution will be documented on the **GC/FID Internal Standard Preparation** worksheet and added to the AgilentFID Quantitation Binder located next to the instrument.

7.4.2.3. An injection of internal standard solution will be made prior to each linear plot determination, each batch of check standard runs, and prior to samples from each case to verify that the internal standard is free of contamination. One injection of the ISTD solution per case file is sufficient. The internal standard blanks will be run on the same method as standards and samples.

7.4.3. Determination of Linearity Plot

7.4.3.1. Five codeine base calibration standards of known concentration (in units of mg/mL) will be prepared over the range of interest using the internal standard solution and will be used to generate the linearity plot. The calibration standards will be labeled with the name and concentration of the solution, date of preparation, and the initials of the analyst who prepared them. The preparation of calibration standards will be documented on the **GC/FID Calibration Standards Preparation** worksheet and added to the AgilentFID Quantitation Binder located next to the instrument.

7.4.3.2. The calibration standard of low concentration will define the method's lower limit of quantitation. The calibration standard of high concentration will define the upper limit of quantitation.

7.4.3.3. Each calibration standard will be injected one time. All instrument conditions must remain constant over the range.

7.4.3.4. A plot of response ratio (calibration standard area/internal standard area) on the y-axis vs. concentration ratio (calibration standard concentration/ internal standard concentration) on the x-axis will be generated using linear regression. The plot of the fit must appear linear. The correlation coefficient (R2) must be greater than or equal to 0.99.

7.4.3.5. If the correlation coefficient is less than 0.99, then appropriate corrective action must be taken. This may include: rerunning the calibration standards, remaking the calibration standards, or performing instrument maintenance.

7.4.3.6. If the correlation coefficient is acceptable, then the completed **GC/FID Linear Plot Calibration** worksheet will be added to the AgilentFID Quantitation Binder located next to the instrument. Document the calibration runs in the instrument logbook.

7.4.4. Check Standards

7.4.4.1. Two codeine base check standards of known concentration (in units of mg/mL) will be prepared within the linear calibration range using the internal standard solution. One check standard should be in the upper calibration range and one check standard should be in the lower calibration range. The check standards should be labeled with the name and concentration of the solution, date of preparation, and the initials of the analyst who prepared them. The preparation of check standards will be documented on the **GC/FID Check Standards Preparation** worksheet and added to the AgilentFID Quantitation Binder located next to the instrument.

7.4.4.2. Both check standards will be analyzed following the determination of a linear plot and once daily prior to instrument use for sample analysis. Both check standards will be injected three times.

7.4.4.3. The concentration of each check standard injection will be calculated using the linear regression equation from the linear plot.

7.4.4.4. The mean and % relative standard deviation (% RSD) of the concentrations will be calculated for both of the check standards. The % RSD (the precision) must be equal to or less than 10% for both check standards.

7.4.4.5. The % difference (accuracy) of the mean from the known concentration (theoretical) of both check standards will be calculated. Each value (the accuracy) must be equal to or less than 10%.

$$\%\ \text{difference (Accuracy)} = \frac{\text{Calculated} - \text{Theoretical}}{\text{Theoretical}} * 100$$

7.4.4.6. If the % difference and % RSD of the check standard concentrations do not meet the listed criteria, then appropriate corrective action must be taken. This may include: rerunning the check standards, remaking the check standards, recalibrating the Linear Plot, or performing instrument maintenance.

7.4.4.7. If both check standards pass the precision and accuracy requirements, the completed **GC/FID Calibration Check** worksheet will be added to the AgilentFID Quantitation Binder located next to the instrument. Document in the instrument logbook that the check standards Passed.

7.4.5. General

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

7.4.5.2. Method parameters are available by the instrument or are electronically retrievable. The data and calculations for each linearity plot and for the check standard determinations will be maintained with the instrument.

**7.5. Procedure**

7.5.1. Preparation of Sample(s)

7.5.1.1. Transfer a known volume of liquid sample into an appropriate disposable container.

7.5.1.2. Add saturated sodium carbonate or 20% sodium carbonate to the sample to make it basic (pH may be checked with pH paper).

7.5.1.3. Add a known volume of internal standard solution. Mix thoroughly.

7.5.1.4. Remove a portion of the internal standard (which now contains the extracted codeine) for analysis by GC/FID.

A second portion of the internal standard extract may be used for analysis by GC/MS. If this is done, then an internal standard solution blank will be run on the GC/MS to demonstrate that it is free from contamination (one run of the internal standard solution per case file is sufficient).

7.5.1.5. Document the volume of sample and internal standard on the **GC/FID Codeine Quantitation** worksheet.

7.5.2. Sample Analysis

7.5.2.1. Analyze the prepared sample extract using the GC/FID by running three replicate injections. Document the sample runs in the instrument logbook.

7.5.2.2. The concentration of each sample injection will be calculated using the linear regression equation from the linear plot.

7.5.2.3. The mean and % relative standard deviation (% RSD) of the concentrations will be calculated for the sample runs. The % RSD (the precision) must be equal to or less than 10% for the sample runs to be acceptable.

7.5.2.4. If the % RSD of the sample runs do not meet the listed criteria, then appropriate corrective action must be taken. This may include rerunning the sample or preparing a new sample extract.

7.5.2.5. If the sample runs pass the listed criteria, then the concentration of codeine in the original sample is calculated using the mean of the sample extract runs, the volume of original sample used, and the volume of ISTD used. The concentration in mg/mL and in mg/100mL is noted on the **GC/FID Codeine Quantitation** worksheet. This worksheet is to be included in the case file.

7.5.2.6. The chromatograms for each sample run, the internal standard run, and the corresponding solvent blank runs as well as the appropriate quantitation reports will be printed, labeled with the unique case identifier, item designators, date, and analyst's handwritten initials and will be maintained with the case file.

**7.6. Interpretation**

7.6.1. The linear plot calibration produces an equation which can be used to determine the concentration of codeine present in a sample. The equation is

$$y = mx + b$$

*where* $y$ = *peak area of codeine / peak area of ISTD*
$m$ = *slope of the line*
$x$ = *concentration of codeine / concentration of ISTD*
$b$ = *Intercept*

solving the equation for the unknown concentration of codeine yields

$$\text{Concentration of codeine (in extracted sample)} = \frac{[\,y - b\,]\,[\,\text{ISTD concentration}\,]}{m}$$

The values of m and b are determined by the linear plot, the value of ISTD concentration is known and included in the method, and the value of y is determined from the sample runs.

7.6.2. For each sample run, the instrument will calculate and report the concentration of codeine in mg/mL. This value will be used to determine the concentration of codeine (mg/mL) in the original liquid sample using the following equation:

$$\text{Concentration of codeine (in original liquid sample)} = \frac{[\text{conc codeine in extract sample}]\,[\text{volume ISTD}]}{[\text{volume original liquid sample used}]}$$

7.6.3. To report the concentration of codeine per 100 mL, multiply the previous value times 100.

**7.7. Estimation of the Uncertainty of Measurement (UM)**

7.7.1. An estimation of the UM is determined for the quantitation of codeine in liquids as this value is being used to fulfill statutory requirements of the Texas Health and Safety Code Section 481.105.

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

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

7.7.4. The final values for the estimation of the UM will be determined in units of mg/100mL to correspond to the units of the reported concentration of codeine (see 7.6.3).

7.7.5. The calculations and final values for the estimation of the UM will be included in the case file.

7.7.6. If the values for the estimation of the UM for a case sample are critical (meaning that the upper limit value could result in an amount of codeine more than 200 mg/100mL), then they will be included on the final report. A statement of the level of confidence such as "Measurement uncertainty of purity measurements are reported at a 95.45% level of confidence" will also be included on the report in these cases.

**7.8. Limitations**

7.8.1. Two or more compounds, especially those with similar chemical structure, can have the same retention time under identical GC conditions.

7.8.2. If a co-eluting component masks the peak of interest, it can interfere with quantitation. It may be possible to resolve the problem by varying the GC program parameters.

7.8.3. Standards of known purity must be used.

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

**7.9. Advantages**

7.9.1. GC provides a good technique for separating components in a mixture and allows quantitation of complex mixtures.

7.9.2. Simple sample preparation is usually sufficient.

7.9.3. A GC auto-sampler increases the efficiency of analysis of numerous samples while functioning unattended.

7.9.4. Samples containing complex mixtures can be quantitated.

**7.10. Literature and Supporting Documentation**

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

7.10.2. L.S. Ettre, Basic Relationships of Gas Chromatography, Perkin-Elmer Corporation, 1977.

7.10.3. A.C. Moffat editor, ”Gas Chromatography,” Clarke’s Analysis of Drugs and Poisons, 3rd edition, (London: The Pharmaceutical Press, 2004) pp.425-499.

7.10.4. SWGDRUG, Measurement Uncertainty for Purity Determinations in Seized Drug Analysis Supplemental Document SD-4, Revision 0, 2013-01-10.

**7.11. Related Documents**

7.11.1. AgilentFID Quantitation Binder

7.11.2. GC/FID Internal Standard Preparation

7.11.3. GC/FID Calibration Standards Preparation

7.11.4. GC/FID Linear Plot Calibration

7.11.5. GC/FID Check Standards Preparation

7.11.6. GC/FID Calibration Check

7.11.7. GC/FID Codeine Quantitation

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

### 8.1. Scope

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

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

8.3.4. Microliter syringe (where applicable)

### 8.4. Standards, Controls, and Calibration

8.4.1. 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.

8.4.1.1. The instrument will be tuned weekly when in use according to the manufacturer's specifications and may be tuned more frequently as deemed necessary.

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

8.4.2. A standard check mix will be injected daily to verify instrument performance when in use. 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 the 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.

8.4.3. 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.

8.4.4. 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.

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).

8.4.6. 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.

**8.5. Procedure**

8.5.1. GC/MS Operating Conditions

8.5.1.1. 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.

8.5.1.2. 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).

8.5.2. Sample Preparation and Analysis

8.5.2.1. Extract samples into a suitable solvent before they are injected into the instrument.

8.5.2.2. Evaluate and print the results of the GC/MS analysis for samples and corresponding blank runs and include the following in the case file:

8.5.2.2.1. The complete Total Ion Chromatogram (TIC) for each sample and corresponding blank run. Evaluated peaks on the TIC will be labeled with the identification of the corresponding mass spectra or "NAM". Analyst discretion will be used when selecting peaks for evaluation based on analytical scheme and circumstances of the case.

8.5.2.2.2. Mass spectra for all evaluated peaks on the TIC.

8.5.2.2.2.1. Mass spectra for identified peaks will include documentation of the comparison of the unknown mass spectra to a known standard, either a stored library comparison or a literature source. If a literature source is used for comparison, the source will be cited in the case file.

8.5.2.2.2.2. Mass spectra for peaks with no known reference comparison (labeled on the TIC as "NAM") will be printed and also labeled as "NAM". The printout may be done manually or as part of a method's automatic data analysis.

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

8.5.2.2.2.4. If a background subtraction is performed for a peak mass spectrum, then retain a copy of the original mass spectrum with the case file as well as the background subtracted mass spectrum. Note the retention time used to generate the background subtracted spectrum on the printout.

8.5.2.2.3. Each page printed will be labeled with the unique case identifier and examiner's handwritten initials and will be maintained with the case file.

Spectra or notes should have the item designators, date, and method of sample preparation (if not listed on the **Controlled Substances Examination Sheet**).

**8.6. Interpretation**

8.6.1. 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.

8.6.2. If used for comparison, results from library searches must be printed and retained with the sample spectra.

**8.7. Limitations**

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

8.7.2. 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.

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

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

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

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

**8.9. Literature and Supporting Documentation**

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

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

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

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

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

**8.10. Related Documents**

8.10.1. Instrument Logbook

8.10.2. Controlled Substances Examination Sheet

## 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 MSDS 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. Sample Analysis

9.5.2.1. Spectra are generally collected and printed with a resolution of at least 4 $cm^{-1}$ scanned from 4000 $cm^{-1}$ to 600 $cm^{-1}$ versus absorbance. This allows comparison to libraries with the same format. Spectral peaks should be of sufficient intensity to make an accurate comparison to known standards or published spectral data.

9.5.2.2. Each spectrum will be printed, labeled with the unique case identifier and examiner's handwritten initials and will be maintained with the case file. Spectra or notes should have the item designators, date, and method of sample preparation (if not listed on the **Controlled Substances Examination Sheet**).

9.5.2.3. Document the comparison of the unknown spectra with a known standard and indicate the source of the known standard in the case file (published or otherwise lab generated).

9.5.2.4. 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.

9.5.2.5. If the straight line function is used to remove interfering peaks from CO2, 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. Interpretation**

9.6.1. 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.

9.6.2. If used for identification, results from library searches must be printed and retained with sample spectra.

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

**9.7. Limitations**

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

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

9.7.3. 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.

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

**9.8. Advantages**

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

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

9.8.3. 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.

**9.9. Literature and Supporting Documentation**

9.9.1. *FT-IR Spectrometer Validation*, Thermo Nicolet Corp., Madison WI, 2001.

9.9.2. "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.

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

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

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

**9.10. Related Documents**

9.10.1. Instrument Logbook

9.10.2. Controlled Substances 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 MSDS 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 solution for the sample

10.3.3.1. Acidic solutions, such as $^{2}/_{3}$ N $H_2SO_4$

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. Spectrophotometer Operating Conditions

10.5.1.1. 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. Sample Preparation

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

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

10.5.3. Sample Analysis

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

10.5.3.2. 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.

10.5.3.3. Each spectrum will be printed, labeled with the unique case identifier and examiner’s handwritten initials and will be maintained with the case file. Spectra or notes should have the item designators, date, and method of sample preparation (if not listed on the **Controlled Substances Examination Sheet**).

**10.6. Interpretation**

10.6.1. The spectra obtained are evaluated with reference to documented sources or spectra from known drug standards. The interpretation of spectra may be reflected directly on the spectrum and will be documented on the **Controlled Substances Examination Sheet** in the appropriate category.

**10.7. Limitations**

10.7.1. 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.

10.7.2. 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).

10.7.3. 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.

**10.8. Advantages**

10.8.1. The test is quick and easy to perform.

10.8.2. Usually very little sample preparation is required.

10.8.3. 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 (diluents) and adulterants.

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

**10.9. Literature and Supporting Documentation**

10.9.1. Sandor Gorog, Ultraviolet-Visible Spectrophotometry in Pharmaceutical Analysis (CRC Press, 1995).

10.9.2. A. F. Fell, “Ultraviolet, Visible, and Fluorescence Spectrophotometry”, Clarke’s Isolation and Identification of Drugs, Second Edition, (London: The Pharmaceutical Press, 1986), 221-236.

10.9.3. 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.

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

10.9.5. Terry Mills III and Conrad J. Roberson, Instrumental Data for Drug Analysis, (New York: Elsevier Science Publishing Co., Inc., 1987).

**10.10. Related Documents**

10.10.1. Instrument Logbook

10.10.2. Controlled Substances 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 and comparison sources and libraries.

### 11.2. Quality Control Procedures for Drug Standards

11.2.1. Drug standards available in the Controlled Substances section will be documented on a **Controlled Substances 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.).

11.2.2. Drug standards will be stored in a securely locked cabinet or refrigerator that can be accessed only by persons authorized by the Section Manager. These provisions are not intended to prevent analysts from having access to small quantities of drug standards at the bench for routine use in analysis.

11.2.3. Before using a new drug standard, an FTIR or GC/MS will be performed to verify that the compound is what it is purported to be. The verification will be documented as part of the **Controlled Substances 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.

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

11.2.5. Any in-house samples will be thoroughly analyzed and characterized before they are used as a drug standard.

11.2.6. If a compound cannot be purchased and is obtained from another forensic laboratory or from a pharmacist (new prescription drugs), then the identity of the substance must be

confirmed by FTIR and/or GC/MS before it can be used as a standard. The verification data will be retained in the section.

11.2.7. An annual inventory of the available drug standards will be conducted and recorded on the **Controlled Substances Drug Standard Usage Log** in conjunction with the **Controlled Substances 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 **Controlled Substances Drug Standard Usage Log** and the **Controlled Substances 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. Physicians 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. Controlled Substances Drug Standard Usage Log

11.4.2. Controlled Substances 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 MSDS 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).

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 Stock Reagents

12.4.3. Monthly Quality Check for Frequently Used Chemical Spot Test 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 MSDS 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 Koppanyi, 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 **Controlled Substances 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 **Controlled Substances 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 **Controlled Substances 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 Controlled Substances 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. Controlled Substances Examination Sheet

**13.10. Koppanyi Test**

13.10.1. Reagents/Chemicals

- Cobalt nitrate, $Co(NO_3)_2 \bullet 6\ H_2O$
- Isopropylamine
- Methanol

*1% Cobalt Nitrate Reagent*: Dissolve 8.0 g $Co(NO_3)_2 \bullet 6\ H_2O$ in 500 ml methanol.

*5% Isopropylamine Reagent*: Add 5 ml isopropylamine to 95 ml methanol.
(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. (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 ($H_2SO_4$)
- 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 $H_2SO_4$.
13.12.2.2. Add one drop of formaldehyde solution.
13.12.2.3. Record any resulting color reactions.

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 color indicates the possible presence of fentanyl.

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 yellow color with the concentrated acid indicates the possible presence of diphenhydramine or methylenedioxy cathinones such as methylone, butylone, pentylone, or MDPV.

13.12.3.8. Formation of a red color indicates the possible presence of salicylates (Aspirin).

13.12.3.9. Formation of a black color upon the addition of the concentrated H2SO4 then orange with fizzing upon the addition of formaldehyde solution (due to the release of NO2) indicates the possible presence of a nitrite.

13.12.3.10. Formation of a dark red color indicates the possible presence of toluene.

13.12.3.11. A yellow powder which forms a deep purple color with the addition of the concentrated H2SO4 followed by a change to yellow with the addition of the formaldehyde solution indicates the possible presence of tetracycline.

13.12.3.12. Some benzodiazepines such as diazepam form an orange color after several minutes.

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 (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. BasicTraining for Forensic Drug Chemists, U.S. Dept. of Justice, 3rd 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
- 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, $FeCl_3 \bullet 6\ H_2O$
- Purified water

*5% Ferric Chloride Reagent*: Dissolve 8.3 g $FeCl_3 \bullet 6\ H_2O$ 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 ($H_2SO_4$)

*Liebermann's Reagent*: Carefully add 5 g sodium nitrite to 50 ml concentrated $H_2SO_4$ 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
- Concentrated hydrochloric acid
- Chloroform
- Petroleum ether

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

Quality-test with a known marihuana sample.

13.21.2. Procedure

13.21.2.1. Place a small amount of plant material in a testing container. Either proceed to the next step or extract the plant material with petroleum ether. If extracted, discard the plant 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 plant 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 the components (cannabinoids, including THC) unique to marihuana, marihuana residue, or hashish 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 MSDS 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. 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/$NH_4OH$ (100:1.5) – general screening
15.3.5.2. T7 – Benzene/Dioxane/95% EtOH/$NH_4OH$ (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 use. 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 used to test the solvent systems and indicator reagents at the same time as casework. The standard will be analyzed with all case samples and a comparison of the $R_f$ values documented on the **Controlled Substances Examination Sheet**.

**15.5. Procedure**

15.5.1. In general, the following steps are taken when analyzing 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.

15.5.1.8. Compare the location of the sample spot to that of the standard.

15.5.1.9. Document the solvent system used to analyze the samples and the results of analysis noting the standards used for comparison on the **Controlled Substances Examination Sheet**.

**15.6. Interpretation**

15.6.1. A positive determination is made when a spot(s) of an unknown substance matches the color and location of the standard.

**15.7. Limitations**

15.7.1. 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.

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

**15.8. Advantages**

15.8.1. TLC is a relatively quick and easy technique.

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

15.8.3. It requires no expensive instrumentation.

**15.9. Literature and Supporting Documentation**

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

15.9.2. A.C. Moffat, “Thin-Layer Chromatography” in Clarke’s Isolation and Identification of Drugs, 2nd edition (London: the Pharmaceutical Press, 1986), 160-177.

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

15.9.4. Miller, J. A.; Neuzil, E. F., Organic Chemistry, Concepts and Applications, (D.C. Heath & Company, Lexington, Mass., 1979), 555.

15.9.5. “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.

15.9.6. “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.

15.9.7. 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.

15.9.8. 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.

15.9.9. 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.

**15.10. Related Documents**

15.10.1. Reagent Logbook

15.10.2. Controlled Substances Examination Sheet

## 16. Excess Quantity Cases

### 16.1. Scope

16.1.1. To provide guidelines for handling excess quantity controlled substance cases.

### 16.2. Policy

16.2.1. An excess quantity case is defined as any controlled substance 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. All excess quantity controlled substance cases will be analyzed by two analysts.

Note: If a latent print examination is requested, the analyst should consult with the latent Print lab and the Section Manager, Supervisors, or designee regarding the handling and transfer of evidence for processing.

### 16.3. Procedure

16.3.1. The receiving analyst and his/her co-worker should place the unique case identifier and 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 Controlled Substances 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, 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 other 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, 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 Controlled Substances 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 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.

Reporting Results of Controlled Substances and Dangerous Drugs

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.*

19.3.2.2. Precede the name of all substances identified with the word **"Contains"**. 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 **Controlled Substances 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 Marihuana, Marihuana Seeds and Hashish

19.3.3.1. Report plant substance identified as marihuana as **"Marihuana"** (not **"contains marihuana"**) and report the net weight in ounces or pounds.

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

*Example:* *Contains Marihuana **

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

19.3.3.3. Report the results of the charred remains of marihuana (from pipes, stubs, ashtrays, etc.) as **"Marihuana"** and the weight as **"trace"** if identified and a residue amount is present.

19.3.3.4. For cases that consist of marihuana seeds only, they may be reported as **"Marihuana seeds"** and the weight in ounces or pounds. If no seeds germinate, report as "**No Controlled Substance Identified**" with a footnote**: "Marihuana seeds were identified and determined to be incapable of beginning germination".**

19.3.3.5. Report hashish and liquid extracts as **"Contains tetrahydrocannabinol"** and the weight in grams.

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 derivate 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 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. Except for marihuana, 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. If marihuana weights are determined in metric units, they will be converted to ounces or pounds for the report. For marihuana samples weighing less than one pound, report the weight of marihuana in ounces. Marihuana samples weighing more than one pound are typically reported in pounds to at least one decimal place instead of in ounces. If a marihuana sample weighs less than 0.01 ounces, the analyst will report the weight as "**Less than 0.01 ounces**".

19.4.5. If the charge for controlled substances identified as belonging in Penalty Group 2-A (synthetic cannabinoids) is delivery, then the weights should be reported in metric units as for Penalty Group 2 substances. If the charge is possession, then the weights should be reported in ounces or pounds as for marihuana. Alternatively, the weights may be reported in both units.

*For example:*

*Bag with plant substance 3.5 grams / 0.12 ounces Contains JWH-018*

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

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 quantitation is performed, then report the determined concentration in the appropriate units and include an appropriate footnote.

*Example: Contains codeine (43.2 mg / 100 ml)**

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

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 (possibly due to insufficient sample), 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*

** Insufficient sample for quantitation*

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 retained with no analysis."*

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. 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, 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
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)
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
II ..... Insufficient ions
Ind ..... Indication
Inv ..... Investigation
ISTD ..... Internal Standard
JIMS ..... Justice Information Management System
juv ..... juvenile
kg ..... Kilograms

lb ..... 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
$R_f$ ..... Retention factor (TLC)
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
DBZP ..... 1,4-Dibenzylpiperazine
dhy ..... Dihydrocodeinone
DMS ..... Dimethylsulfone
GBL ..... gamma-butyrolactone
GHB ..... gamma-hydroxybutyric acid(γ-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 Controlled Substances 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 which may be the Controlled Substances Section Manager, the Quality Assurance Manager or Centralized Evidence Receiving staff.

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 **Controlled Substances Reanalysis Form**.

## 22.4. Related Documents

22.4.1. Controlled Substances Reanalysis Form

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

**24. Modification Summary**

| ISSUE DATE | CHANGE |
|---|---|
| 12-01-16 | Modifications to this version include but are not limited to the following changes:<br>Updates to safety and personal protective equipment throughout<br>The requirement for maintenance and quality checks to be documented in a logbook is replaced throughout with the requirement that documentation be maintained within the section<br>Section 14. Chemical Microcrystalline Tests rescinded and references throughout SOP removed<br>1.2 Objectives updated<br>2.2 Submission of Evidence section removed<br>2.2.2 Inventory of evidence and handling of discrepancies updated<br>3.3.2 References to active cases removed<br>3.3.2.9 – 3.3.2.10 Updates to communications regarding item selection<br>3.4.6.4 Notation of statistical sampling on the Exam Sheet removed<br>3.5.3.2.2 Documentation of sample preparation for GC/MS included<br>3.5.5 Remove specific weight cut-off values<br>3.5.6 Clarification of weight variation for volatile liquids<br>3.6.2 Add “A good rule of thumb is to use less than half of the total sample.”<br>4.2 Requirement for all case folder documents to be labeled with the unique case identifier added and the requirement for initials on court orders and communication records removed.<br>4.7.2 Inclusion of photographs as examination documents specifically added<br>5.2 Use of “Neg” defined for chemical spot tests ; Weighing Events/Uncertainty – “…These values are required ~~only determined~~ for substances…”<br>5.3 Use of the Controlled Substances Notes Sheet clarified<br>6.7.4 -6.7.5 and 6.7.8 Balances and weights will be calibrated/certified/checked annually<br>6.7.7.2 Determination of the acceptance range for balance checks modified<br>6.8.3-6.8.4 Pipette check criteria updated |

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| | 6.10.4 Equipment Service Form added<br>8.4.5 Vial septa bleed added as possible source of blank chromatogram peaks<br>8.4.6 "…would be within 30 days ~~the same month~~ as long as…"<br>8.5.1.2 "These lists will be updated annually ~~once a year~~ or more frequently…"<br>9.5.2.5 Requirements for use of the straight line function with the FTIR added<br>11.2.3 Location field on the Drug Standard Verification Log clarified<br>12.3.1.1 and 13.4.1 Concentration added to reagent labeling<br>12.3.3.2 and 12.3.5.2 Labeling for aliquots added<br>13.4.2 Koppanyi added as frequently used reagent<br>13.7.3 Negative clarified for chemical spot tests<br>13.12.3.2 Formation of an orange color in the Marquis test indicative of fentanyl<br>16.4.1 Section for retention of samples added<br>16.4.2.1 Amount of plant substance to retain in excess quantity cases changed<br>18.3.2 – 18.3.3 Frequency for estimation of the UM for weight determinations clarified<br>19.4.4 Reporting of marihuana weights over one pound clarified<br>20. Abbreviations for "gr", "net", "wh", "env", "evi", "APAP" added or clarified<br>23. Guidelines for Processing Non-Active Cases rescinded and references throughout SOP removed |
| 10-23-15 | Entire document format including font, margins, numbering, etc. modified for standardization.<br>Additional modifications to this version include the following changes:<br>Delete 3.5.6 "An approximate volume should be determined for liquids containing phencyclidine and codeine."<br>Add to Technical Review 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."<br>Delete under 5.2.2 "**Approximate Volume** – The approximate volume will be noted for liquids identified as containing codeine or phencyclidine. The approximate volume for other liquids may also be noted." |

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| | 5.4 Marihuana Checklist updated to note the stereoscope used for observing sample characteristics.<br><br>6.7.7.2 Acceptable ranges for balance types updated and criteria for determining acceptable ranges modified following annual UM evaluation.<br><br>Add 6.7.7.3 "…and that the weight is centered on the pan…"<br><br>Add 6.7.8.4 "…and that the weight is centered on the pan…"<br><br>Modify 7.6.3 "…multiple the previous value…" to "…multiply the previous value…"<br><br>Modify 18.3.10 "…in the same units as the reported weight." to "…in the same units as the recorded weight."<br><br>Delete "and Volumes" from 19.4<br><br>Modify 19.5.1 "Report the number of abuse units of LSD samples as defined…" to "Report the number of abuse units for substances identified as belonging to Penalty Group 1-A as defined…"<br><br>20.2 Add UM = Uncertainty of Measurement to General Abbreviations |
| 09-01-15 | Modifications to this version include the following changes:<br><br>Sections 6.4.2, 9.4.1, and 9.4.3 updated to reflect the replacement of Val-Q performance check software for the FTIR with ValPro software resulting from an overall software update from OMNIC 7.1 to OMNIC 9.3. |

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