

HOUSTON POLICE DEPARTMENT



CRIME LABORATORY DIVISION CONTROLLED SUBSTANCES SECTION STANDARD OPERATING PROCEDURES

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01 GOALS AND OBJECTIVES

The primary goal of the Controlled Substances Section is to support the mission of the **Crime Laboratory** by providing quality analysis of evidence received for the presence of controlled substances, dangerous drugs, and other chemical substances as efficiently as possible utilizing available resources.

To maximize efficiency, cases submitted will be reviewed and the case status identified as Active (grand jury or court requests, priority investigations, cases pending in court), Non-Active (court accepted plea, submitted for destruction, charges dismissed by the court), or as not having a clear status. Evidence associated with Active cases becomes the primary focus of the section and will be handled based upon the following objectives:

- All priority items should be analyzed as soon as they are received and completed before the end of the day.
- Botanical cases (live plants) should be dried as soon as possible once received by an analyst and analyzed within one week once dried.
- All active excess quantity controlled substance cases should be analyzed within two weeks.
- Fifty percent of all active controlled substance cases should be analyzed and completed within two weeks.
- Seventy-five percent of all active controlled substance cases should be analyzed and completed within thirty days.
- All active controlled substance cases should be analyzed and completed within sixty days.
- All reports should be generated as soon as possible after the completion of a case but within two working days.
- All case files should be technically and administratively reviewed within five working days following the generation of the report.
- All evidence should be returned to Centralized Evidence Receiving (CER) within five working days of the completed case file review.

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- The time between receipt of evidence by an analyst and the return of that evidence to CER should be less than one month. If evidence cannot be returned to CER within 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.)
- See the Guidelines for Processing Non-Active Cases Section for processing of these and unclear status cases.

02 EVIDENCE HANDLING

SCOPE

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

SUBMISSION OF EVIDENCE

Narcotic evidence may be submitted for analysis to the following places:

- Through the offsite lock boxes at various locations
- In person to the laboratory through Centralized Evidence Receiving

EVIDENCE HANDLING BY CENTRALIZED EVIDENCE RECEIVING (CER)

CER personnel receive controlled substance evidence into the laboratory, enter case related information into the Evidence Management System (EMS), and store the evidence until it is transferred to an analyst. When the analyst has completed work on a case, the evidence is returned to CER personnel to be handled according to CER SOP.

RECEIVING EVIDENCE

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.

All evidence received by a drug analyst is to be assigned by the Section Manager or designee and must be documented as follows:

- (1) Each proximal container (bag, envelope, box, etc.) must be marked with a unique case identifier (either the assigned incident number or laboratory number) and the analyst's initials. The proximal container is usually a Houston Police Department Evidence Envelope, but it can be anything that contains exhibits for a case. In addition, an item designator may be used with the incident or laboratory number to distinguish items within a case.
- (2) A submission form must be filled out for all evidence submitted. If the officer has not attached a submission form to the evidence, a submission form is filled out with all pertinent information available.

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- (3) All exhibits should be inventoried and compared with the documentation on the submission form. The analyst will itemize the actual evidence received on the Controlled Substances Examination Sheet. **If there are significant discrepancies between the submission form and the exhibits themselves, such as missing exhibits, notify your supervisor immediately.** Generally, these cases will be investigated in-house with notification going to the submitting officer, Narcotics command, and the Laboratory Director if necessary. A supplement should be entered into OLO by the assigned analyst documenting the facts surrounding the case at the conclusion of the investigation.
- (4) The information on the submission form should match the information written on the evidence envelope. If there is a discrepancy, such as mismatched suspect names or different incident numbers, an effort should be made to clarify the information through resources such as OLO, EMS, LIMS and/or JIMS before reporting the results of analysis. The discrepancy may simply be the result of writing or typing the information incorrectly or the submitting officer may have inadvertently switched forms with other evidence. It is sometimes necessary to contact the officer to determine the cause of the discrepancy and/or to have the officer come to the laboratory to correct the situation. **Always notify your supervisor about major discrepancies or if assistance is needed in correcting discrepancies.**
- (5) All exhibits contained within a case should be labeled with the analyst's initials and the unique case identifier and item designators. In a case with numerous small items analyzed 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 the screening tests a difference is noted, then the small items will be grouped appropriately and analyzed and labeled separately.

CASES CONTAINING CURRENCY, VALUABLES, LARGE ITEMS, AND BULLETS

- (1) All U.S. currency, valuables, large items, and bullets should be prepared by the analyst for transfer to the Property Room by CER personnel, unless they are returned to the submitting officer. 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. In a case with numerous bills, recording the serial numbers may be suspended. 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) Evidence to be transferred to the property room should be noted as such on the report of analysis.

CASES REQUIRING EXAMINATION FOR LATENT PRINTS

Generally, latent print requests are made on the submission form when evidence is submitted for analysis. In addition, police officers or Assistant District Attorneys (ADA's) 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 informs the person making the request that the evidence has already been handled so that the requestor can determine if prints are still needed.

Regardless of how or when a latent print request is made, the analyst will remove packaging to be printed and prepare it for transfer to the Latent Print section. 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.

CASES CONTAINING POSSIBLE BIOHAZARDS

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).

Some items that require special handling are the following:

- (1) **Syringes** - remove any needles with the needle cutters.
- (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.

- (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.

SUBMITTING EVIDENCE TO CER

- (1) Repackage all evidence in the same condition it was received whenever possible. If chunk substance is found in a matchbox inside a ziplock, repackage it as you found it. Do not change the condition of the evidence unless it is absolutely necessary. For example, liquid in an open soda can will be transferred to a jar that can be sealed.
- (2) Before sealing evidence for submission to CER, double check that all evidence is properly labeled.
- (3) Seal evidence (including analyst initials and date) according to the Crime Laboratory QA and SOP and submit to CER.

03 ANALYSIS GUIDELINES

SCOPE

To provide guidelines for the analysis of controlled substances and dangerous drugs.

PROCEDURE

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.

The general guidelines for which items in an active case need analysis are as follows (see the Objectives Section for the description of active cases):

- 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.
- If the charge is Tampering and only residues are present then at least one residue per suspect should be analyzed.
- 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).
- 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.
- 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.
- Any items in a case indicated as being seized due to a delivery transaction should be analyzed.
- If there are multiple suspects listed on the submission form, it may be necessary to analyze more items than those outlined above. Check all sources of information.

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- In each case, the most significant items should be identified and analyzed. Considerations must be given to the information provided on the Evidence Submission Form, or available through OLO and JIMS. 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.
- Items which are not analyzed will be documented and reported as "Retained with no analysis".
- If an analyst consults with the officer, Assistant District Attorney, or an intern 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. Document the conversation and maintain the documentation with the case file.
- In all cases, request for analysis of unanalyzed items by a principal associated with a case may require further analysis of retained items.

BASIC ANALYTICAL SCHEME (POWDERS, TAR, AND CHUNK SUBSTANCE)

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.

- One confirmatory instrumental test (either FTIR or GC/MS) and at least one other different positive test (including chemical screening tests, microcrystalline, TLC, UV/VIS, 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.
- If a case contains multiple containers of powder, tar, or chunk substance that are similar in appearance (appearance refers to the actual powder, etc., not similar packaging), then sufficient containers will be sampled to ensure that the highest weight limit is surpassed. Containers which are not sampled will be documented and reported as retained with no analysis.
- A weight is determined and recorded for all powders, tar, and chunk substance to be reported. If the weight is at a cut-off weight (i.e., 1.0 grams, 4.0 grams etc.), then the next significant figure other than zero is recorded and reported. The balance used to determine the weight shall be indicated on the Examination Sheet. It is the analyst's

responsibility to verify that the balance they use conforms to the laboratory's calibration guidelines.

- Data required for instrumental analyses

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 that the data is observed must be recorded on the Examination Sheet. The following should also be documented:

- (1) **UV**
All appropriate information regarding sample preparation, wavelengths, weights, absorbances, or calculations should be documented on the UV graph or in the notes.
- (2) **GC/MS**
All appropriate information regarding retention times and library matches should be documented on the GC/MS graph(s) or in the notes. A graph of the blank run prior to the sample should be maintained with the case file.
- (3) **FTIR**
All appropriate information regarding sample preparation and library/literature searches should be documented on the FTIR graph or in the notes.
- (4) **GC/FID**
All appropriate information regarding sample preparation, retention times, weights, or calculations should be documented on the GC/FID graph(s) or in the notes. A graph of internal standard runs should be maintained with the case file. A graph of blank runs prior to the internal standard and sample runs should be maintained with the case file.

Maintenance and quality assurance procedures are documented and available by each instrument. It is the analyst's responsibility to verify that an instrument is working properly before use.

- Non-instrumental methods may be used to aid in the analysis of powders, tar, and chunk substance. These methods may include thin layer chromatography, microcrystalline tests, and chemical screening tests:

- (1) Thin Layer Chromatography
Each solvent system used is listed on the Examination Sheet. The observations are documented as well as the standards used for comparison.
- (2) Microcrystalline Tests
Each reagent system used is listed on the Examination Sheet. The observations are documented. This documentation may be either a written description of the microcrystal or a drawing. In addition the performance of blank controls is documented on the Examination Sheet.
- (3) Chemical Screening Tests

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

LIQUIDS

- If a case contains multiple containers of liquid that are similar in appearance, then sufficient containers will be sampled to ensure that the highest weight limit is surpassed. Containers which are not sampled will be documented and reported as retained with no analysis.
- A weight and volume should be determined on all liquids to be reported except those in an abusable volatile chemical case. In those cases, an estimated volume may be recorded.
- It is common for phencyclidine (PCP) liquids to evaporate rapidly (ether based solvent) so PCP cases should be analyzed on a priority basis. Because of this evaporation, the weight obtained by the analyst may be less than the weight listed by the officer.
- For remaining analysis follow the analytical scheme given under powders, tar, and chunk substance.

TABLETS AND CAPSULES - GENERAL

- 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.

- 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.
- 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. Tablets and/or capsules which are not analyzed will be documented and reported as retained with no analysis.
- A weight and number should be determined and recorded for all controlled substance tablets or capsules that will be reported. If dangerous drug tablets or capsules are reported, no weight is necessary. If the total number of tablets in one grouping is too numerous to count, then an approximate number is determined and noted. It is acceptable to describe the number of tablets as numerous instead of approximate on the report.

PHARMACEUTICAL TABLETS AND CAPSULES

- The first step in attempting to identify tablets and/or capsules is to compare their markings (logo) with reference materials. If they are successfully identified as pharmaceutical products, this is considered to be an acceptable screening test. When performing a pharmaceutical identification, a hardcopy (e.g. computer printout or xerox copy) should be included in the case file as well as documentation of the source. The markings (logos) observed by the analyst should be noted on the Examination Sheet for comparison. All attempts at identification, even those that are unsuccessful should be documented on the Examination Sheet.

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.

While partial logos can give useful information as to the possible identity of a pharmaceutical product, they cannot be used as a test for identification. Noting the results of partial logo searches on the Examination Sheet is acceptable as long as this is not used as a test. In this case follow the analytical scheme for Clandestine Tablets and Capsules.

- When pharmaceutical identification is successful, only one tablet or capsule from each grouping needs to be fully analyzed by performing a confirmatory test (GC/MS or FTIR).
- 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 should be followed.

CLANDESTINE TABLETS AND CAPSULES

- 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. The analytical scheme given under powders, tar, and chunk substance should be followed.
- For clandestinely manufactured tablets or capsules, a sufficient number should be sampled for the appropriate weight limit (based on the substance identified and the charge). Tablets (capsules) which are not sampled will be documented and reported as retained with no analysis.
- For each grouping of tablets (capsules) to be reported, each item up to 29 should be 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.
- For large numbers of clandestine tablets or capsules an alternative sampling plan which may be used is to select random samples to prove a statistically significant number of the items are positive for a controlled substance or dangerous drug. One method of random sampling relies on the theory of hypergeometric distribution. For a population (grouping) of 1000 or more tablets, randomly selecting 29 tablets from the population is sufficient to statistically conclude with a 95% certainty that 90% of the population contains the substance identified in the selected random sample. To use statistical sampling to make conclusions regarding a population the analyst should perform the following steps:
 - (1) Determine the total number of items in the population (grouping) to be sampled and record the total weight of the population.
 - (2) After selection, each item is to be analyzed separately and completely.
 - (3) If presumptive testing indicates a difference in the randomly selected items, then all items in the population (grouping) will need to be analyzed separately

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or the population will need to be subdivided into separate groups as appropriate.

- (4) Documentation should be noted on the Examination Sheet that statistical sampling was used.
- If the analyst has any questions regarding the sampling or analysis of clandestine tablets (capsules) he/she should consult with the Lab Manager or designee.

DRUG RESIDUES

- 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). Residues which are not sampled will be documented and reported as retained with no analysis.
- A small amount of the residue is removed for analysis, ensuring that enough residue remains for an independent analysis. If the amount of residue is too small to retain a sufficient sample for reanalysis, then procedure blanks should 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 should be included in the case notes.

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

- Instrumental and non-instrumental methods may be used to aid in the analysis of residues (follow the analytical scheme given under powders, tar, and chunk substance).
- If visual examination of evidence which is needed for charges (e.g. one suspect has a pipe and another suspect has only a push rod) indicates that no sample/residue is present for analysis (the push rod), then the item should be examined by another analyst to confirm the lack of sample. Both analysts will initial the observation on the Examination Sheet. The item is to be reported as “No analysis performed (no visible sample)”.
- When field testers are received without any other evidence to analyze, they should be reported as “No unprocessed sample available for analysis.” If requested to analyze

the field tester, then the analyst should document the requestor information (name, phone number, and position) and handle appropriately depending on the amount of sample present for analysis (i.e. is there enough sample available for reanalysis).

PLANT SUBSTANCE AND PLANT SUBSTANCE RESIDUES

- Samples should be taken from each container (bag, cigar, cigarette, etc.) of plant substance analyzed. If there are multiple containers, then sufficient containers will be sampled to ensure that the highest weight limit is surpassed. If the containers are cigarettes or cigars, then a sample should be taken from the middle of the item. Containers which are not sampled will be documented and reported as retained with no analysis.
- Live plants:
 - (1) Plants are dried before weighing and analyzing.
 - (2) Remove roots, dirt and mature stalks before weighing (mature stalks = thick stalk ~1 centimeter in diameter or larger which test negative for THC content)
 - (3) The weight for the dried plants will be significantly less than the officer's listed weight.
- A macroscopic and microscopic analysis is performed on all samples taken. Any features found are documented on the Marihuana Checklist. For the identification of marihuana a minimum of 2 microscopic characteristics should be observed including cystolithic hairs or glandular hairs.
- Microscopic identification and at least one other positive test (including the Duquenois / Duquenois-Levine chemical screening test or GC/MS) are required for the identification of marihuana, excluding seeds.
- Germination of Marihuana Seeds:

Seeds are identified by color and appearance. The viability of seeds may be determined by germinating a sample of the seeds.

 - (1) Select seeds for germination.
 - (2) Place seeds between moistened filter paper or the equivalent, and place in an appropriate container.
 - (3) Incubate at room temperature for up to 10 days.
 - (4) Document the number of seeds that germinated.

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

- For mushrooms or plant material suspected of containing psilocin / psilocybin the Weber chemical screening test may be performed to test for the presence of psilocin / psilocybin. If the Weber test is positive a positive confirmatory test (GC/MS or FTIR) must be performed to report the presence of psilocin / psilocybin.
- 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 marijuana using microscopic and chemical screening tests (see the analytical scheme under powders, tar, and chunk substance).
- A weight is determined and recorded on all plant substance items that are analyzed including cigars, cigarettes, cigar stubs, and cigarette stubs. The weights determined for cigars and cigarettes should not include the weight of the wrapper (paper or tobacco leaf). At least one cigar or cigarette should be opened completely to determine the appropriate wrapper weight to subtract from the total sample weight. If cigar stubs and cigarette stubs need to be analyzed, the weight of the paper may be included in the total weight and this is to be indicated both on the report and on the Examination Sheet. If the weight of the cigarette stubs or cigar stubs makes a difference to the weight cut-off in the law, then the paper should be removed. Pipes and residues are not weighed. If marijuana weights are determined in metric units, they will be converted to ounces or pounds for the report.
- In cases where plant substance has undergone excessive decomposition, the item should be examined by another analyst and both analysts will initial the observation on the Examination Sheet. The item is to be reported as “No analysis performed due to excessive decomposition”.
- 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). The plant substance from non-manufactured items in such cases should be tested to determine if it is marijuana.

NO CONTROLLED SUBSTANCE IDENTIFICATION

- Before an item can be reported as “No Controlled Substance Identified”, a GC/MS sample should be run.
- 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.
- If an initial GC/MS sample run is negative (no measurable peaks in the Total Ion Chromatogram), then a second more concentrated sample should be run. This can be achieved either by the use of additional sample or by evaporation of the initial sample if no more sample is available. The analyst should document sample preparation steps in the case file.
- If the peaks present in a GC/MS sample run do not indicate the presence of a controlled substance or they are identified as being non-controlled substances (e.g. lidocaine, caffeine), then the item may be reported out as “No Controlled Substance Identified” without an additional GC/MS sample run. However, if a controlled substance peak is indicated but cannot be positively identified, then a second more concentrated sample should be run as described above.
- If the only substance(s) identified by FTIR are non-controlled (e.g. lidocaine, caffeine) or cannot be identified, then GC/MS testing should be performed before reporting the results to ensure that a controlled substance is not being masked.

LITERATURE AND SUPPORTING DOCUMENTATION

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

04 CASE DOCUMENTATION

SCOPE

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

CONTENTS OF CASE FOLDER

- Report on the results of the analysis which has been technically and administratively reviewed and includes the analyst's name, title, and signature.
- Laboratory Evidence Submission Form and any other submission forms (e.g. Latent Lab, Property Room) or chain of custody records in printed or electronically retrievable format.
- Laboratory Examination Sheet(s) 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. Each Examination Sheet must have the unique case identifier (HPD incident number or laboratory number) and item designators, the date for each observation and/or test, and the analyst's handwritten initials.
- Analytical Data
 1. All charts, spectra, notes, and photographs 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.
 2. All solvent blanks run prior to any case samples for the **GC/FID or GC/MS** should be maintained with the case file.
- Any court orders or Motions for Discovery (labeled with the unique case identifier on each page and the initials of the analyst complying with the court order or Motion for Discovery).
- A record of all pertinent phone calls (labeled with the unique case identifier and initials).

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ADMINISTRATIVE REVIEW

All case folders will be administratively reviewed by an individual other than the author of the report prior to issuance of the report. An administrative review should include the following:

- Verify that the incident number provided is the correct incident number for the case being entered.
- Verify all weights entered. It is very important to verify that the weights entered match the weights on the Examination Sheet since this information is used to charge the suspect. The weight information on the submission form should also be checked to ensure that the analyst has not put the wrong designation, such as milligrams instead of grams.
- Verify all spelling, grammar, the unique case identifier and item designators, and the analyst's name and employee number. Results from all pages of the Examination Sheets should be included in the report.
- Verify that the correct information is listed for the evidence submitted.
- The completed administrative review is indicated on the final report with the reviewer's name, title, and signature. The date of the completed administrative review is electronically documented in a retrievable format.

TECHNICAL REVIEW

All case folders will be technically reviewed. This review should include the following:

- Verify that the weights on the report match the weights on the Examination Sheet. Check that the weights on the submission form are consistent with the reported weights.
- Verify that all spectra support the conclusion.
- Verify that all spectra contain the appropriate unique case identifier and item designators.

- Verify that all spectra contain any pertinent documentation and that the spectra are documented on the Examination Sheet. Check for the presence of any necessary blanks.
- All Examination Sheets and spectra must have the analyst's handwritten initials.
- Verify that all observations listed on the Examination Sheet are consistent with the conclusion(s).
- The completed technical review is indicated on the final report with the reviewer's name, title, and signature. The date of the completed technical review is electronically documented in a retrievable format.
- Before giving any verbal results to a requestor (example priority, rush, investigation, etc) the analysis should be technically reviewed. This can be documented in the case file on an activity log by having the person performing the technical review sign and date. The results of analysis still need to be included in the official report.

ADMINISTRATIVE AND TECHNICAL REVIEW ISSUES

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 Laboratory Manager or designee, in person, as soon as possible. Administrative review issues should also be reported if it becomes a pattern.

REPORT MODIFICATION RECORDS

- 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.
- If it becomes necessary to amend a signed report, then the incorrect report must be documented so as not to be confused with the corrected report. It is recommended that a single line be drawn through the incorrect information. The initials of the employee making the change must also be included. The original, corrected report must be maintained within the case record. If a new report is issued, the new report will be uniquely identified, will contain a reference to the original report that it replaces and should clearly state why an amended report was issued.

05 EXAMINATION SHEET

SCOPE

To provide guidelines for documentation of tests and observations on the Examination Sheet.

EXAMINATION SHEET

- The first line is documented with the unique case identifier (HPD incident number or laboratory number), date, and analyst's initials. The second line is documented with the item designators used to distinguish items within the case. All observations are dated appropriately. For example, if a case is started and completed with all observations on the same day, then the date listed at the top is appropriate for all observations and tests. Any observations on a different date than the start date should be documented accordingly.
- Document all evidence appropriately including the outer sealed evidence containers in the space provided for "EVIDENCE SUBMITTED". Notations and descriptions of evidence should be clear to a reviewer of the case file. Acceptable abbreviations which may be used in describing evidence are included in the Abbreviations Section and at the bottom of the Examination Sheet. All documented evidence should be identified with an item designator including items which are not analyzed. The exception to the documentation of evidence on the Examination Sheet and the assignment of item designators includes copies of submission forms, in-take forms, pieces of paper with officers' initials, etc.
- A modified version of the Examination Sheet is available for use in involved cases to help clarify evidence descriptions. When this modified Examination Sheet is used it should be the first page of examination documentation with a notation made on each subsequent page in the "ITEM" line to refer to the first page for the description of evidence. Item designators may be used on subsequent pages to refer to evidence.
- When testing two or more items under one "test" (spot tests, microcrystalline, etc.), place the number of items and the observation.
- When analyzing marijuana, the microscopic box, the Duquenois box and the Duquenois-Levine box are filled out with the number of samples tested and the observations. **Pos** may be used in the microscopic box to indicate that characteristics for marijuana were observed in the sample. These characteristics should be documented on the Marijuana Checklist. If no characteristics of marijuana are observed microscopically, then **Neg** may be used in the microscopic box on the

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Examination Sheet and the appropriate box checked on the Marijuana Checklist; alternatively, a notation such as “no characteristics of marijuana observed” may be used in the microscopic box in place of using the Marijuana Checklist.

- Chemical screening tests (spot tests) are documented by noting the observations and number of samples tested in the appropriate boxes.
- Spot plates are to be visually examined for cleanliness by the analyst prior to use. A check mark on the Examination Sheet next to “Spot Plate Check” indicates that the spot plates used were free of residue or debris.
- The reagent system used for any microcrystalline tests is documented along with the observations.
- Blank (or negative) controls for all chemical screening tests (including the Duquenois and Duquenois-Levine) as well as for all microcrystalline 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.
- UV tests, **GC/FID tests**, GC/MS comparisons, and FTIR comparisons are documented appropriately.
- The solvent system used to run thin layer chromatography plates is documented along with the observations as well as the standard(s) used for comparison. If UV visualization is used to visualize the TLC plate this should be noted.
- Information obtained from pharmaceutical identifications (PHI) such as the DEA Logo Search, the PDR (*Physician's Desk Reference*) or other references is recorded appropriately in the PHI/VISUAL box. Any unsuccessful pharmaceutical identification attempts are also recorded. The markings (logos) observed by the analyst should be noted for comparison.
- Items which do not appear to have residue present may include the notation “no visible sample” or “NVS” in the PHI/VISUAL box.
- For items in which the entire submitted sample is used for analysis but a portion of the sample remains once the analysis is complete, the Examination Sheet should be documented appropriately.

For example: *“All evidence used for analysis, remaining portion retained with the case.”*

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- For items where there is no sample remaining after analysis, the Examination Sheet should be documented appropriately.

For example: *“All evidence used for analysis, no remaining sample available.”* or use the abbreviation *“EDIA”* which stands for *“Evidence Destroyed in Analysis”*.

- Notations regarding the condition of the evidence when received should be included on the Examination 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.
- Measurable weights and volumes are recorded appropriately. The analyst will document on the Examination Sheet which balance is used for any weight determination.
- When significant quantities of evidence are consumed during analysis, it is recommended that before and after analysis weights be noted on the Examination 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.
- The results of the analysis which are to be reported are noted in the space provided for "RESULTS". If the results are negative, then "NCS" or "NCSI" is written. If an item is not subjected to analysis then, "Retain" is noted in the space provided.
- When a case is reopened and further analysis is required, the following procedures should be followed when the original Examination Sheet is used:
 1. The date of any additional testing is documented appropriately.
 2. If the additional testing is performed by a different analyst, then his/her initials are documented appropriately.

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

06 INSTRUMENT PERFORMANCE AND MAINTENANCE

SCOPE

To establish quality assurance guidelines for the maintenance, performance, and repair of analytical instrumentation and balances.

GENERAL REQUIREMENTS FOR ANALYTICAL INSTRUMENTATION

All instruments will be periodically maintained and their performance verified in accordance with the manufacturer's recommendations and specifications and **Crime Laboratory** policy. All instruments' performance 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. Refer to the Crime Laboratory QA and SOP for the guidelines regarding retention of performance verification records.

UV/VIS SPECTROPHOTOMETER

- Conduct a performance verification check on UV/VIS instruments quarterly or as needed.
- 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 485.7 nm - 486.3 nm and 655.8 nm - 656.4 nm respectively.
- Standards will also be used to verify that the instrument is performing as expected. To do this weigh three samples of one of the validated standards (currently methamphetamine, heroin, or cocaine) and perform a quantitation using the experimentally determined E value. The determined purity should be within 10% of the expected value.
- 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.
- Maintain a logbook with the results.

FTIR SPECTROMETER

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

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- One method is to use the OMNIC Val-Q software to check the performance of the instrument. The measurements made by Val-Q are designed to meet a subset of the specifications contained in ASTM Standard Practice E1421-99 and utilize two polystyrene samples, one 1.5 mil thick and one 3.0 mil thick. Val-Q tests the spectrometer's single-beam energy ratio, 100% line peak-to-peak noise, 100% line root-mean-square (rms) noise, 1.5 mil polystyrene band position, resolution factor, and 3.0 mil polystyrene zeros. Factory-based limits for each test are coded into Val-Q so that pass-fail results can be reported.
- Maintain a logbook with the results.

GAS CHROMATOGRAPHY/FLAME IONIZATION DETECTOR (GC/FID)

Performance Verification Check

- When using GC for quantitation, the instrument will be calibrated using a series of known concentrations of a standard.
- 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%.
- 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.
- Maintain a logbook with the results

Other GC/FID Maintenance

- Run a solvent blank before all other runs and maintain a copy of the blank run with the case file.
- Perform regular and preventive maintenance according to the manufacturer's recommendations. A logbook documenting all non-routine maintenance (e.g., column replacement and any major repairs) will be kept with the instrument.

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GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Performance Verification Check

- The Mass Selective Detector (MSD) should be tuned weekly when in use or more often as needed (e.g. if the instrument is moved or maintenance is performed on the MSD).
- The instrument should be tuned according to the manufacturer's instructions and must meet the manufacturer's specification.
- A standard should be run daily when in use and the scan results entered in the 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.
- Maintain a logbook with the results.

Other GC/MS Maintenance

- Run a solvent blank before each sample run and maintain a copy of the blank run with the case file.
- Perform regular and preventive maintenance according to the manufacturer's recommendations. A logbook documenting all maintenance (e.g. column replacement, filament replacement, seal replacement, vacuum oil changes, source cleaning, and major repairs) will be kept with the instrument.

BALANCES

- Laboratory personnel will check balances for accuracy regularly, using standard weights. Balances must be checked whenever they are moved from one location to another. Laboratory standard weights should be checked after the annual re-certification of the balance.
- Balances will be certified by an external vendor at least once a year.
- Inspect the balances for cleanliness and check the level frequently.
- The appropriate balance will be used for the weight being measured and precision required. Care should be taken not to overload a balance with too much weight.

- Since the tolerances of electronic balances vary, the instrument specifications must be checked to determine the appropriate criteria for satisfactory performance.
- The following general specifications may be used:

<u>Balance class</u>	<u>Weights</u>	<u>Significant figures</u>	<u>Acceptable variation</u>
Analytical	4.0 g	4.0000 g	±0.0001 g
	1.0 g	1.0000 g	±0.0001 g
	5.0 mg	0.0050 g	±0.0001 g
Top Loading	2.0 kg	2000.0 g	±0.1 g
	2.0 kg	2000.00 g	±0.01 g
	1.0 kg	1000.0 g	±0.1 g
	1.0 kg	1000.00 g	±0.01 g
	4.0 g	4.0 g	±0.1 g
	4.0 g	4.00 g	±0.01 g
	1.0 g	1.0 g	±0.1 g
1.0 g	1.00 g	±0.01 g	
Bulk Scale	4.0 kg	4.00 kg	±0.02 kg
	4.0 kg	4000 g	± 1g
	2.0 kg	2.00 kg	±0.02 kg
	2.0 kg	2000 g	± 1g

Other standardized weights may be used at the analyst's discretion.

- Analytical balances should be checked with standard weights at least weekly.
- Top loading balances should be checked with standard weights monthly or as needed.
- The bulk scales should be checked with standard weights prior to use.
- Maintain a logbook with the results of the balance checks, standard weight checks, maintenance, and certification.
- It is the analyst's responsibility to verify that the necessary checks have been performed in the recommended time period for any balances or standard weights used.

Malfunction of an Instrument or Balance

- If an instrument or balance fails the performance check or a performance problem is detected during routine maintenance, it must be removed from service, the section manager or designee must be notified and the problem recorded in the logbook.
- No instrument or balance is to be used if it is not in proper working order.
- Repair or have the instrument or balance repaired and perform routine quality control procedures with standards to ensure it is working properly before the instrument or balance is returned to service. Verification with standards will be performed after routine maintenance if the performance of the instrument could be affected.
- Keep a record of all repairs and routine maintenance in a logbook.

REFRIGERATION

- Refrigerators and freezers are used for the storage of heat sensitive chemicals, standards, and reagents. They should be monitored at least once a week to ensure that they are working properly and that refrigerators are within 2°C to 8°C and that freezers are below 0°C. A record is to be maintained documenting the date checked, the displayed temperatures, and the initials of the individual performing the check.
- If the temperature should fall outside of the acceptable range, verify that the unit has power and that air circulation has not been impeded. If corrective action does not return the unit to normal operation, then notify the section manager or designee. A technical representative may need to be called for service or the refrigerator may need to be replaced.

07 GAS CHROMATOGRAPHY (GC) FOR QUANTITATION OF CODEINE IN LIQUIDS

SCOPE

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).

SAFETY

- Use appropriate safety equipment when preparing reagents and handling volatile chemicals. Refer to the MSDS for additional safety information for specific chemicals.
- Properly secure high-pressure gas cylinders.
- Use caution around hot surfaces.
- Discard all chemicals and any other pertinent materials in an appropriate manner.

EQUIPMENT, MATERIALS, AND REAGENTS

- Gas chromatograph equipped with a flame ionization detector
- Auto-sampler vials and caps
- Injection syringe
- Analytical balance needed for quantitation
- Pipettes and Dispenser
- Suitable solvents for sample preparation
- Codeine reference standard
- Octacosane (C28) hydrocarbon to be used as internal standard

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STANDARDS, CONTROLS, AND CALIBRATION

A valid five point linearity plot using codeine base reference 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.

Internal Standard Solutions

- 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.
- 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.
- 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.

Determination of Linearity Plot

- 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.
- 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.

- Each calibration standard will be injected one time. All instrument conditions must remain constant over the range.
- 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 (R^2) must be greater than or equal to 0.99.
- 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.
- 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.

Check Standards

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

- 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%.

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

- 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.
- 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.

General

- 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.
- 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.

PROCEDURE

Preparation of Sample(s)

- Transfer a known volume of liquid sample into an appropriate disposable container.
- Add saturated sodium carbonate or 20% sodium carbonate to the sample to make it basic (pH may be checked with pH paper).
- Add a known volume of internal standard solution. Mix thoroughly.
- 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).

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

Sample Analysis

- Analyze the prepared sample extract using the GC/FID by running three replicate injections. Document the sample runs in the instrument logbook.
- The concentration of each sample injection will be calculated using the linear regression equation from the linear plot.
- 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.
- 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.
- 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.
- 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.

INTERPRETATION

- 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 $y = \text{peak area of codeine} / \text{peak area of ISTD}$

$m = \text{slope of the line}$

$x = \text{concentration of codeine} / \text{concentration of ISTD}$

$b = \text{Intercept}$

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.

- 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:

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

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

LIMITATIONS

- Two or more compounds, especially those with similar chemical structure, can have the same retention time under identical GC conditions.
- 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.
- Standards of known purity must be used.
- The peak to be quantitated must be a single component peak and completely resolved.

ADVANTAGES

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

- Simple sample preparation is usually sufficient.
- A GC auto-sampler increases the efficiency of analysis of numerous samples while functioning unattended.
- Samples containing complex mixtures can be quantitated.

LITERATURE AND SUPPORTING DOCUMENTATION

- D.A. Skoog and J.J. Leary, *Principles of Instrumental Analysis*, Saunders College Publishing, 1992, pp. 432,434, 622.
- L.S. Ettre, *Basic Relationships of Gas Chromatography*, Perkin-Elmer Corporation, 1977.
- A.C. Moffat editor, "Gas Chromatography," *Clarke's Analysis of Drugs and Poisons*, 3rd edition, (London: The Pharmaceutical Press, 2004) pp.425-499.

08 GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

SCOPE

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

SAFETY

- Use appropriate safety equipment when preparing reagents and handling volatile chemicals. Refer to the MSDS for additional safety information for specific chemicals.
- Properly secure high-pressure gas cylinders
- Use caution around hot surfaces such as oven interiors and injection and detector ports.
- Discard all chemicals and any other pertinent materials in an appropriate manner.

EQUIPMENT, MATERIALS, AND REAGENTS

- Gas chromatograph/mass spectrometer analytical instrument
- Auto-sampler vials and caps
- Solvent(s) appropriate for the substance being analyzed
- Microliter syringe (where applicable)

STANDARDS, CONTROLS, AND CALIBRATION

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

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2. Tune records are maintained in a file in the laboratory. If the tune is not successful, the instrument will be taken out of service until corrective action is taken.
- A standard should be injected daily to verify instrument performance when in use. The standard printout should be maintained with the appropriate tune report. If the standard run does not provide an acceptable mass spectral identification, the instrument should be retuned and the standard rerun. If the standard still does not provide an acceptable mass spectral identification, then the instrument will be taken out of service until corrective action is taken and the problem recorded in the logbook.
 - Solvent blanks will be injected between case samples to verify that the column and syringe are free of contamination. The solvent blank will be run on the same method as the sample and immediately before it.
 - A procedure blank will be run for samples that will be completely consumed by analysis to verify that the column, reagents, solvents, and laboratory glassware used are clean prior to the analysis of case samples. A procedure blank for GC/MS analysis should 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 should be included in the case notes. If any sample remains after analysis, then the procedure blank vials and sample vials used should be evaporated to dryness, labeled appropriately, and retained with the case evidence.
 - Any significant peaks in the blank chromatograms should be properly investigated to identify their source (e.g. column breakdown, carryover from previous sample run, or instrumental contamination) so that corrective action 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).
 - 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 the same month as long as instrument conditions had not changed (column replacement or method modifications). Available and verified standards are a requirement for this practice.

PROCEDURE

GC/MS Operating Conditions

- Use appropriate temperature programs and adjust other critical parameters to ensure that the suspected substance(s) will elute during data collection. The program should allow a reasonable time for unknown or unexpected compounds to elute.
- 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 should be updated once a year or more frequently as needed (for example following column changes or method modifications).

Sample Preparation and Analysis

- Extract samples into a suitable solvent before they are injected into the instrument.
- Print and retain the charts depicting the results of the GC/MS analysis in the case file. Include the following:
 1. The complete Total Ion Chromatogram (TIC) for each sample and corresponding blank run.
 2. Mass spectra for all peaks corresponding to controlled substances and/or other substances of interest
 3. 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.
 4. Document the comparison of the unknown spectra to a known reference, either a stored library comparison or a literature reference. If a literature reference is used for comparison, cite the source.
 5. Each page 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 examination sheet).

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INTERPRETATION

- Library searches can be used to provide useful information pertaining to the identity of a compound but should not be used as a replacement for verifying positive identification based on spectral peaks.
- If used for comparison, results from library searches must be printed and retained with the sample spectra.

LIMITATIONS

- When analysis by GC/MS is unable to provide positive identification, another technique (FTIR, derivatization, etc.) must be utilized to provide positive identification.
- 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.
- It may be difficult to identify individual compounds in a homologous series.

ADVANTAGES

- Generally, mass spectra of compounds of interest are specific to single compounds and may be used for positive identification.
- It may be possible to separate and identify complex mixtures that are difficult to separate through ordinary clean-up procedures.
- The technique is useful for analyzing small sample amounts that may be difficult to identify using other techniques.
- A GC/MS auto-sampler increases the efficiency of analysis of numerous samples by functioning unattended.

LITERATURE AND SUPPORTING DOCUMENTATION

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

- F. W. McLafferty, *Interpretation of Mass Spectra*, 4th Edition, (Sausalito, California: University Science Books, 1993).
- Jehuda Yinon, *Forensic Mass Spectrometry*, (Boca Raton, Florida: CRC Press, Inc., 1987).
- J. Throck Watson, *Introduction to Mass Spectroscopy: Biomedical, Environmental, and Forensic Applications*, (New York: Raven Press Books, 1140 Avenue of the Americas, 1976).
- R. E. Ardrey, “Mass Spectrometry” in *Clarke’s Isolation and Identification of Drugs*, (London: The Pharmaceutical Press, 1986), 251-263.

09 FOURIER TRANSFORM INFRARED (FTIR) SPECTROMETRY

SCOPE

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

SAFETY

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

EQUIPMENT, MATERIALS, AND REAGENTS

- Fourier transform infrared spectrometer
- Mortar and pestle (if needed)
- Attenuated Total Reflectance (ATR) accessory
- Acetone or suitable solvent (for cleaning)

STANDARDS, CONTROLS, AND CALIBRATION

- A performance verification check should be performed quarterly or more often as needed and recorded in an appropriate logbook. One method is to use the OMNIC Val-Q software to check the performance of the instrument. The measurements made by Val-Q are designed to meet a subset of the specifications contained in ASTM Standard Practice E1421-99 and utilize two polystyrene samples, one 1.5 mil thick and one 3.0 mil thick. Val-Q tests the spectrometer's single-beam energy ratio, 100% line peak-to-peak noise, 100% line root-mean-square (rms) noise, 1.5 mil polystyrene band position, resolution factor, and 3.0 mil polystyrene zeros. Factory-based limits for each test are coded into Val-Q so that pass-fail results can be reported.
- 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 corrective action is taken.

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- The test results obtained by utilizing the Val-Q performance checks are compared to prior results to verify that the system is working consistently over time.
- A background will be taken before each sample scan and this step is included in the experimental method used for sample analysis.

PROCEDURE

Sample Preparation

- 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.
- 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:
 - (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.
 - (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.

Sample Analysis

- 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 reference libraries with the same format. Spectral peaks should be of sufficient intensity to make an accurate comparison to known reference standards or published spectral data.

- 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 Examination Sheet).
- Document the comparison of the unknown spectra with a known reference and indicate the source of the reference in the case file (published or otherwise lab generated).
- 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.

INTERPRETATION

- Library searches can be used to provide useful information pertaining to the identity of a compound but should not be used as a replacement for verifying positive identification based on spectral peaks.
- If used for comparison, results from library searches must be printed and retained with sample spectra.
- The infrared spectrum of the majority of controlled substances and other substances routinely identified is specific to a single compound and may be used for identification.

LIMITATIONS

- The sample must be relatively pure for positive identification.
- For an accurate comparison of an unknown spectrum to a standard spectrum, both samples (the sample and the reference) must be in the same salt form. Some compounds may produce different crystal structures that can result in slightly different infrared spectra.
- Infrared cannot usually be used to distinguish between optical isomers.

ADVANTAGES

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

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

LITERATURE AND SUPPORTING DOCUMENTATION

- *FT-IR Spectrometer Validation*, Thermo Nicolet Corp., Madison WI, 2001.
- “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.
- Fell, A. F., *Clarke’s Isolation and Identification of Drugs*, (London: The Pharmaceutical Society of Great Britain, 1986).
- *Forensic Science Handbook*, Volume III, ed. By Richard Saferstein, (Englewood Cliffs, N.J.: Regents/Prentice Hall, 1993).
- Skoog, D. A., *Principles of Instrumental Analysis*, 3rd Edition, (New York: Saunders College Publishing, 1985) 148-149.

10 ULTRAVIOLET/VISIBLE SPECTROPHOTOMETRY (UV/VIS)

SCOPE

A nondestructive technique for the preliminary identification of controlled substances, dangerous drugs and other substances. To establish a procedure to determine the concentration of a controlled substance, dangerous drug, or other substance in a sample using ultraviolet spectrophotometry. Details about UV/VIS quantitation will be given after the general information section.

SAFETY

Use appropriate safety equipment when preparing reagents and pouring liquids. Refer to the MSDS for additional safety information for specific chemicals. Dispose of all chemicals in an appropriate manner.

EQUIPMENT, MATERIALS, AND REAGENTS

- UV/VIS spectrophotometer
- Quartz cuvettes, matched pair, or equivalent
- An appropriate solution for the sample
 1. *Acidic solutions, such as $\frac{2}{3}$ N H₂SO₄*
 2. *Basic solutions, such as 0.45 N NaOH*
 3. *Methanol or ethanol*
- Analytical balance

STANDARDS, CONTROLS, AND CALIBRATION

- A UV/VIS performance verification check should be performed quarterly or as needed and recorded in an appropriate logbook. 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 485.7 nm - 486.3 nm and 655.8 nm - 656.4 nm respectively.

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- For comparison purposes, refer to reliable published reference materials, analyze known control samples, or refer to in-house spectral collections produced from known samples.
- Reference solvent blanks should be run at the same time using the same solvent as sample.
- Refer to the end of this section for further information on UV/VIS quantitation.
- 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 corrective action is taken and the problem recorded in the logbook.

PROCEDURE

Spectrophotometer Operating Conditions

- 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.

Sample Preparation

- Dissolve the sample in a solution appropriate for the substance.
- 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.
- Plant materials will require extraction, while mixtures and other substances may require extraction prior to analysis.

Sample Analysis

- Collect a spectrum of the sample in the appropriate solution.
- 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.
- 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 examination sheet).

INTERPRETATION

The spectra obtained are evaluated with reference to documented sources or spectra from known samples. The interpretation of spectra may be reflected directly on the spectrum and should be documented on the Examination Sheet in the appropriate category.

LIMITATIONS

- An ultraviolet spectrum is not specific, and a positive identification cannot be made exclusively on the basis of UV/VIS analysis.
- 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. the dangerous drug carisoprodol has no UV absorption).
- 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.

ADVANTAGES

- The test is quick and easy to perform.
- Usually very little sample preparation is required.
- 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 (dilutents) and adulterants.
- This is usually a non-destructive technique and the sample can be recovered for other testing procedures, if necessary.
- May provide a quick and easy quantitation of some drugs/dilutants/adulterants.

QUANTITATION BY UV/VIS

(quantitative analysis for investigative purposes suspended per 03-03-10 memo)

STANDARDS, CONTROLS AND CALIBRATIONS

- Performance verification check done quarterly or as needed. Standards will be used to verify that the instrument is performing as expected. To do this weigh three samples of one of the validated standards (currently methamphetamine, heroin, or cocaine) and perform a quantitation using the experimentally determined E value. The determined purity should be within 10% of the expected value.
- Reference solvent blank.
- $E_{1\text{cm}}^{0.1\%}$ (E-Value) for the compound of interest (This may be obtained from reference literature or determined/confirmed with laboratory standards on the instrument prior to using this technique for quantitation. If the latter procedure is used, the results will be documented in the appropriate logbook.)
- Controlled substance reference standard for the drug to be quantitated.
- If an instrument fails a performance check or a performance problem is detected during routine maintenance or use, it should be taken out of service until corrective action is taken and the problem recorded in the logbook.

PROCEDURE

Sample Preparation

- Obtain a representative sample of the substance requiring quantitation. The amount needed will vary according to the concentration of the controlled substance in the sample and the E-value or absorptivity of the controlled substance. For best results:
 1. Adjust the concentration of the controlled substance so that the absorbance is strong enough to differentiate the peaks from background noise and yet weak enough to remain in the linear absorbance range.

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2. The sample must be diluted such that the absorbance is within the determined linear range.
- For powdered (solid) samples:
 1. To reduce the effects of the inherent percent error in weighing the sample on the final quantitation results, the analyst should use a larger quantity of the sample and dilute as necessary to obtain a solution that gives an absorbance in the linear range.
 2. Some samples may require extraction before they can be quantitated.
 - For liquid samples:

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

Sample Analysis

- A baseline spectrum should be obtained by scanning the desired wavelength range.
- Collect a spectrum of the sample, subtracting the background at the appropriate wavelength for the drug being analyzed.
- Calculations to determine the concentration should be included in the case folder, either on the UV chart or in the notes.
- 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 examination sheet).

INTERPRETATION

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

$$A = abc, \text{ where}$$
$$A = \text{absorbance value}$$
$$ab = E_{1\text{cm}}^{0.1\%} \text{ value [b = path length = 1 cm]}$$

c = concentration

- Note that the *E*-values in *Clarke's* are at 1.0% and must be divided by 10 in order for the resultant calculation to yield a concentration (c) value of mg/ml (0.1%).
- For basic drugs, report the quantitation results in base form. The concentration as the salt may be reported only if the analyst has identified the salt form by an accepted analytical procedure.

LIMITATIONS

- UV quantitation is not suitable for samples that do not absorb UV light or for those that contain interfering compounds (such as nicotinamide and pseudoephedrine in methamphetamine samples) that modify the absorbance of the sample at the quantitation wavelength.
- This technique is usually not suitable for samples with more than one controlled substance.
- Samples that are not suitable for ultraviolet quantitation may be quantitated using an alternate technique such as gas chromatography.

ADVANTAGES

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

LITERATURE AND SUPPORTING DOCUMENTATION

- Sandor Gorog, *Ultraviolet-Visible Spectrophotometry in Pharmaceutical Analysis* (CRC Press, 1995).
- A. F. Fell, "Ultraviolet, Visible, and Fluorescence Spectrophotometry", *Clarke's Isolation and Identification of Drugs*, Second Edition, (London: The Pharmaceutical Press, 1986), 221-236.
- 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.

- Douglas A. Skoog and Donald M. West, *Principles of Instrumental Analysis* (New York: Holt, Rinhart, and Winston, Inc., 1971).
- Terry Mills III and Conrad J. Roberson, *Instrumental Data for Drug Analysis*, (New York: Elsevier Science Publishing Co., Inc., 1987).

11 STANDARDS AND REFERENCES

SCOPE

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

QUALITY CONTROL PROCEDURES FOR DRUG STANDARDS

- Before using a new drug standard, an FTIR or GC/MS will be performed to verify that the compound is what it was purported to be. The spectra will be placed in a quality control book which will include all pertinent information such as the lot number, source and initials of the analyst who performed the test.
- Some commercially prepared drug standards are mailed with GC/MS and other quality control data. These data sheets will be retained.

VERIFICATION OF STANDARDS THAT CANNOT BE PURCHASED COMMERCIALY

- Thoroughly analyze and characterize any in-house samples before they are used as a standard or reference.
- If a compound cannot be purchased and is obtained from another forensic laboratory that has already encountered the problem 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 reference. The verification data will be retained in the laboratory.

LIBRARY REFERENCES

When analyzing compounds, particularly drugs, using either GC/MS or FTIR, the spectra will be compared to a reference standard. The source of the reference may be an in-house library, a published library (such as NIST), any accepted published reference (such as *Clarke's Isolation and Identification of Drugs*), or a spectrum from a reliable source (such as DEA). The instrument libraries on the FTIR include the Georgia State Library and the in-house library (HPD) generated from standards. The instrument libraries on the GC/MS instruments **include but are not limited to** the American Academy of Forensic Science (AAFS) Library, the National Institute of Standards and Technology (NIST) Library, **the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Library**, and the in-house libraries (HPD) generated from standards.

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12 REAGENT QUALITY ASSURANCE

SCOPE

The following describes quality assurance guidelines for reagents, chemical preparations, and solvents used in drug analysis.

SAFETY

- Use appropriate eye protection and other safety equipment to avoid contact with chemicals.
- Refer to the appropriate MSDS for the safe handling of chemicals.
- Discard all chemicals and any other pertinent materials in an appropriate manner.

PRACTICE

All pertinent reagents and solutions will be labeled with the identity of the reagent and the date of preparation (or lot number). A quality control logbook will be maintained and will include the following information, when applicable:

- Reagent preparation date
- Preparer's initials
- Standard used and the results of a positive quality control check of the reagent
- Results of a negative (blank) quality control check of the reagent
- Initials of the analyst(s) who quality tested the reagent and the date of testing

Quality Testing for Frequently Used Reagents

Frequently used reagents will be quality tested at the time of preparation 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. The new reagent will be quality tested prior to being used and the appropriate information recorded in the logbook. The quality testing should 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.

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.

Aliquots for reagents used 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.

See the Chemical Screening Tests and Microcrystalline Tests Sections for a listing of the current **Frequently** used reagents.

Quality Testing for All Other Reagents

Infrequently used reagents will be quality tested upon preparation 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.

TLC (thin layer chromatography) reagents will be quality tested during use by the analyst using an appropriate standard and the results will be documented in the case notes.

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.

Quality Assurance

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

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:

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

- Discard the reagent if it fails the quality check, prepare a new reagent, and quality check the reagent with a known standard.
- Cease performing casework with these reagents until the problem has been corrected.
- Identify casework that may have been affected by the reagents/chemicals that failed the quality check and re-test with quality checked reagents.
- Inform the Quality Manager if the problem persists.

Record

Logbooks or appropriate documentation.

13 CHEMICAL SCREENING TESTS

SCOPE

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

SAFETY

- Chemical spot tests may use a variety of corrosive, caustic, or other dangerous chemicals. Caution should always be practiced, and appropriate personal protective safety equipment should be used.
- Any solutions that fail a quality check will be discarded in an appropriate manner.
- Refer to MSDS for additional safety information for specific chemicals.

EQUIPMENT, MATERIALS, AND REAGENTS

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

STANDARDS AND CONTROLS

- Each spot test stock reagent must be labeled with the name of the reagent or solution 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.
- The frequently used spot test reagents are Ferricyanide, Marquis, Van Urk's, Cobalt thiocyanate, and Duquenois. These reagents will be quality tested at the time of preparation 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 at the time of preparation and prior to use.
- It is the responsibility of the analyst to quality check infrequently used reagents and document appropriately on the examination sheet. Proper documentation includes

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

- It is the responsibility of the analyst to determine if reagents are working properly. Blank (or negative) controls for chemical screening 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 screening 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.

DEFINITIONS

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

LIMITATIONS

- All spot tests are presumptive in nature and serve only as a guide for an analyst's analytical scheme.
- Adulterants and complex mixtures may produce reactions that interfere with the clear interpretation of the results.
- A sample with a low concentration of a particular substance may yield negative spot test results.

ADVANTAGES

- Spot tests provide a quick and easy method for determining what type of compound or functional group a sample might contain.
- Spot tests can assist in the determination of appropriate analytical processing, collection of appropriate samples, and the grouping of samples for uniformity testing.

INTERPRETATION

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

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- With weak color changes, the analyst may choose to document the color preceded by the designation “weak.”

KOPPANYI TEST

Reagents/Chemicals

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

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

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

Quality-test reagent with a barbiturate standard.

Procedure

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

Interpretation

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

Literature and Supporting Documentation

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

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

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FERRICYANIDE TEST (also known as Simon's test)

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.

Procedure

1. Combine a small amount of sample with a few drops of ferricyanide reagent.
2. Add a few drops of 20% sodium carbonate.
3. Record any observations.
4. The reagent combination itself turns a deep red. This color is the normal color for a negative reaction.

Interpretation

- 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).
- Some secondary amines (MDE, N-OH MDA) do not form a blue color or form only a slight purple color due to steric hindrance.
- Strongly basic solutions will form a deep red color before the addition of the 20% sodium carbonate.

Literature and Supporting Documentation

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

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MARQUIS TEST

Reagents/Chemicals

- Concentrated sulfuric acid (H₂SO₄)
- Formaldehyde solution (~ 37% formaldehyde; stored in the refrigerator)

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

Procedure

1. Combine a small amount of sample with a few drops of concentrated H₂SO₄.
2. Add one drop of formaldehyde solution.
3. Record any resulting color reactions.

Interpretation

- Formation of an orange to brown color indicates the possible presence of amphetamine, methamphetamine or phentermine (other substances may show similar color formations).
- Formation of a purple to black color indicates the possible presence of MDMA, MDE, and MDA.
- Formation of a green to black color indicates the possible presence of dextromethorphan.
- Formation of a green color indicates the possible presence of 2,5-dimethoxyphenethylamine and its derivative 4-bromo-2,5-dimethoxyphenethylamine (Nexus, 2C-B).
- Formation of a purple color indicates the possible presence of heroin, other opiates, methocarbamol, or guaifenesin.
- 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.
- Formation of a red color indicates the possible presence of salicylates (Aspirin).

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- 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.
- Formation of a dark red color indicates the possible presence of toluene.
- 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.
- Some benzodiazepines such as diazepam form an orange color after several minutes.
- There may be other substances that form various colors with the reagents.

Literature and Supporting Documentation

- H.M. Stevens 1986. "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A.C. Moffat (London: The Pharmaceutical Press) pp. 128-147.
- S.H. Johns, et. al. "Spot Tests: A Color Chart Reference for Forensic Chemists," *Journal of Forensic Sciences* 24 (1979) pp. 631-649.
- 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.
- K. E. Toole, et. al. "Color Tests for the Preliminary Identification of Methcathinone and Analogues of Methcathinone," *Microgram Journal* 9(1), pp. 27-32.

VAN URK'S TEST

(also known as p-Dimethylaminobenzaldehyde or Erlich's Test)

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.

Procedure

1. Combine a small amount of sample and a few drops of Van Urk's reagent.
2. Record any observations.

Interpretation

- Formation of a bright yellow color indicates the possible presence of primary aromatic amines such as procaine and benzocaine.
- 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).
- 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).

Literature and Supporting Documentation

- H.M. Stevens, 1986. "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A.C. Moffat (London: The Pharmaceutical Press) pp. 128-147.
- S.H. Johns, et. al. "Spot Tests: A Color Chart Reference for Forensic Chemists," *Journal of Forensic Sciences*, 24 (1979): pp. 631-649.
- Basic Training for Forensic Drug Chemists, U.S. Dept. of Justice, 3rd edition.

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

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COBALT THIOCYANATE / MODIFIED COBALT THIOCYANATE TEST

(Cocaine test; Scott's test)

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.

Procedure

1. Combine a small amount of sample with the cobalt thiocyanate reagent.
2. If a color change is observed, record any observation.
3. Add one drop of concentrated hydrochloric acid.
4. Add a few drops of chloroform to extract any soluble complexes.
5. Record any observations.

Interpretation

- 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.
- 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.
- The cobalt thiocyanate test is a useful step in distinguishing cocaine salt from cocaine base.
- 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.

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Literature and Supporting Documentation

- L.J. Scott, "Specific Field Test for Cocaine," *Microgram* 6 (1973): pp. 179-181.
- 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.
- 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.
- S.H. Johns, et. al. "Spot Tests: A Color Chart Reference for Forensic Chemists," *Journal of Forensic Sciences* 24 (1979) pp. 631-649.
- 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.

JANOVSKY TEST

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.

Procedure

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

Interpretation

- Formation of a purple color indicates the possible presence of diazepam or flunitrazepam.
- 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.
- Formation of a yellow color indicates the possible presence of clonazepam or nitrazepam.
- No color formation is seen with alprazolam or lorazepam.

Literature and Supporting Documentation

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

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WEBER TEST

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.

Procedure

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.
2. Record any observations.
3. Add a few drops of concentrated hydrochloric acid.
4. Record any observations.

Interpretations

- 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.

Literature and Supporting Documentation

- 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.

FERRIC CHLORIDE TEST

Reagents/Chemicals

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

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

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

Procedure

1. Combine a small amount of sample with a few drops of 5% ferric chloride reagent.
2. Record any observations.

Interpretation

- Formation of a red-orange color indicates the possible presence of GHB.
- Formation of a dark purple color indicates the possible presence of salicylates (aspirin).
- Formation of a bluish gray color indicates the possible presence of acetaminophen.

Literature and Supporting Documentation

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

LIEBERMANN TEST

Reagents/Chemicals

- Sodium nitrite
- Concentrated sulfuric acid (H₂SO₄)

Liebermann's Reagent: Carefully add 5 g sodium nitrite to 50 ml concentrated H₂SO₄ 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.

Procedure

1. Combine a small amount of sample and a few drops of Liebermann's reagent.
2. Record any observations.

Interpretation

- Various colors may be formed by a large number of different compounds. Results or interpretations can be found in Stevens (1986).
- A variety of color results for steroids may be found in Chiong (p.491).

Literature and Supporting Documentation

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

SULFURIC ACID TEST

Reagents/Chemicals

- Concentrated sulfuric acid

Quality-test reagent with a steroid standard.

Procedure

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

Interpretation

- Formation of an orange or yellow color may indicate the possible presence of a steroid.
- Formation of a yellow color may also indicate the possible presence of diphenhydramine.

Literature and Supporting Documentation

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

MANDELIN TEST

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.

Procedure

1. Combine a small amount of sample and a few drops of Mandelin's reagent.
2. Record any observations.

Interpretation

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

Literature and Supporting Documentation

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

DUQUENOIS / DUQUENOIS-LEVINE TEST

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.

Procedure

1. Place a small amount of plant material in a testing container.
2. Either proceed to the next step or extract the plant material with petroleum ether.
3. If extracted, discard the plant material and evaporate to dryness.
4. Add one part of the Duquenois reagent and wait approximately one minute.
5. Add one part concentrated hydrochloric acid. (the Duquenois test)
6. Record any observations.
7. Add one part chloroform. (the Levine modification)
8. Record any observations.

Interpretation

- 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 tetrahydrocannabinol (THC).
- Formation of a purple color in the chloroform layer indicates the possible presence of tetrahydrocannabinol.
- 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.

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Literature and Supporting Documentation

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

14 MICROCRYSTALLINE TESTS

SCOPE

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

SAFETY

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

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

EQUIPMENT, MATERIALS AND REAGENTS

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

STANDARDS AND CONTROLS

- Each microcrystalline test stock reagent must be labeled with the name of the reagent or solution 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.
- The frequently used microcrystalline test reagents are aqueous Gold Chloride and aqueous Platinum Chloride. These reagents will be quality tested at the time of preparation and monthly thereafter with the date of preparation and most recent quality testing noted on all in use containers. All other microcrystalline test reagents are considered infrequently used and must be quality checked at the time of preparation and prior to use.
- It is the responsibility of the analyst to quality check infrequently used reagents and document appropriately on the examination sheet. Proper documentation includes noting the reagent used, the standard used, and the results. See the Reagent Quality Assurance Section for further explanation of quality testing procedures.

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- It is the responsibility of the analyst to determine if reagents are working properly. Blank (or negative) controls for microcrystalline 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.

LIMITATIONS

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

ADVANTAGES

- It requires very small amounts of sample for a successful test.
- Most microcrystalline tests are relatively quick, easy, and specific for the compound tested.

PROCEDURES

In general, the following steps are taken when analyzing case samples:

- Dissolve a small portion of the sample in a suitable solvent on a microscope slide.
- Place a small amount of reagent on a cover slip.
- Invert the cover slip and carefully drop onto the microscope slide allowing the reagent and sample solution to mix.
- Observe the formation of characteristic microcrystals under a microscope.

INTERPRETATION

Microscopic observations are documented on the examination sheet by writing a description or drawing of what is observed.

LITERATURE AND SUPPORTING DOCUMENTATION

- E.G.C. Clarke editor, Isolation and Identification of Drugs, Volume 1, 1978. "Microcrystal Tests", pp. 135-141.

15 THIN LAYER CHROMATOGRAPHY(TLC)

SCOPE

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

SAFETY

- 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.
- 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.
- Developing solvents and indicator reagents should be discarded in an appropriate manner.

EQUIPMENT, MATERIALS, AND REAGENTS

- Silica gel on aluminum, glass, polyester, or other appropriate medium
- Glass developing tank
- Capillary tubes, micropipettes, or equivalent
- UV light box (long and short wave)
- TLC solvent systems and developing sprays as outlined in the Training Guide

STANDARDS AND CONTROLS

An appropriate known reference standard should be used to test the system and detection reagents. The standard should be analyzed with all case samples and a comparison of the R_f values documented.

PROCEDURE

In general, the following steps are taken when analyzing case samples:

- Extract the sample with an appropriate solvent.
- 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.
- Allow the sample to dry after application.
- Place the plate vertically into a solvent tray with enough solvent moisture to cover 0.5 to 1.0 cm of the sample end of the plate.
- Allow the solvent front to rise near the top of the TLC plate.
- 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.
- Apply an appropriate indicator spray and/or view under UV light to visualize the component(s) of interest.
- Compare the location of the sample spot to that of the standard.
- Document the solvent system used to analyze the samples and the results of analysis noting the standards used for comparison.

INTERPRETATION

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

LIMITATIONS

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

ADVANTAGES

- TLC is a relatively quick and easy technique.
- It can be used as a clean-up procedure for complex mixtures.
- It requires no expensive instrumentation.

LITERATURE AND SUPPORTING DOCUMENTATION

- Bobbitt, J. M.; Schwarting, A. E.; Gritter, R. J., *Introduction to Chromatography*, 1968.
- A.C. Moffat, "Thin-Layer Chromatography" in *Clarke's Isolation and Identification of Drugs*, 2nd edition (London: the Pharmaceutical Press, 1986), 160-177.
- Fox, R. H.; "Paper Chromatography", in *Isolation and Identification of Drugs*, ed. E.G.C. Clarke (London: The Pharmaceutical Press, 1969), 43-58.
- Miller, J. A.; Neuzil, E. F., *Organic Chemistry, Concepts and Applications*, (D.C. Heath & Company, Lexington, Mass., 1979), 555.
- "Chromatographic Data, Thin Layer Chromatography Tables, Volume I, Sec. II.IV", *CRC Handbook of Chromatography*, Volume I, edited by Robert C. Weast, CRC Press, Division of the Chemical Rubber Company, 1972, 477-487.
- "Practical Applications II.I Detection Reagents for Paper- and/or Thin Layer Chromatography", Volume 2, Section II, *CRC Handbook of Chromatography*, edited by Robert C. Weast, CRC Press, Division of the Chemical Rubber Company, 1972, 103-189.
- E. Buel, C. N. Plum, and S. K. Frisbie, "An Evaluation of a Partition Thin Layer Chromatography System for the Identification of Cannabinoids", *Microgram*, 15 (1982): 145-157.

- 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.
- 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.

16 EXCESS QUANTITY CASES

SCOPE

To provide guidelines for handling excess quantity controlled substance cases.

POLICY

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 or designee regarding the handling and transfer of evidence for processing.

PROCEDURE

- The receiving analyst and his/her co-worker should place the unique case identifier and initials on all exhibits.
- 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 should be labeled to include the unique case identifier and item designators, analysts' handwritten initials, and the date the photos were taken. A videotape may be taken at any time at the discretion of the analyst.
- 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.

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.

- If the total weight for the case is near one of the weights used as a cut-off in the Texas Drug Laws, the receiving analyst will determine the appropriate weighing method.
- 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.).
- 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 **Examination Sheet**.

To determine an appropriate representative sample:

1. The representative sample will consist of a minimum of five separate containers randomly sampled from the total amount of evidence.
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.
3. Evidence that consists of one single container of liquid will require the taking and preserving of only one representative sample.
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 on the worksheet under each item.
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.).
6. If a large excess quantity case is composed of evidence from multiple addresses, retain a representative sample from each source.

- At least one set of initials from all submitting officers, if available, and the receiving analyst or CER representative 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 should be labeled as "**Representative Sample.**"
- The remainder of the case will be packaged as excess quantities as follows:
 1. The container size for excess quantities should be limited to forty pounds. The following information should be on each container:
 - Analysts' initials and unique case identifier;
 - Notations of "**1/5, 2/5, etc.**" or "**1 of 5, 2 of 5, etc.**" or the **EMS item number (001, 002, CER104123, etc.)** to identify multiple containers of the same case; and
 - "**Excess.**"
 2. 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.

RETENTION OF SAMPLES

Excess Quantity Plant Substance:

- Approximately 1.5 kilograms should be retained as a representative sample. At least five separate containers must be present (Health and Safety Code section 481.160).
- 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.
- 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.

Excess Quantity Powders:

- 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.

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

Excess Quantity Liquids:

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

Tablets and Capsules:

- 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.

REPORTING

The report of analysis for an excess quantity case should follow the Reporting Guidelines Section as usual with the inclusion of the following footnote:

The Houston Police Department Crime Laboratory has photographed, determined the total weight of the substances and has retained representative samples as prescribed under the provisions of chapter 481.160 of the Texas Controlled Substance Act. The excess quantities may be destroyed 28 days after separate notification unless the Laboratory receives notice from the District Attorney's office before that date. The Houston Police Crime Laboratory will retain sufficient documentation as to the ultimate disposition of the narcotic(s).

SUBMISSION TO CER

- The case folder (including photos) must be technically reviewed prior to its final submission to CER.
- The analyst will submit the entire case to the Centralized Evidence Receiving (CER) section for storage utilizing the following instructions:

1. The representative sample and the case folder will be personally delivered to CER personnel.
2. CER personnel will personally verify all portions of the case to be stored, both the representative sample and the excess quantities.

17 CLANDESTINE LABORATORIES

This Section is rescinded as of August 16, 2004.

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18 MONTHLY INVENTORY

SCOPE

A monthly inventory of most of the controlled substances received in the laboratory is sent to the Department of Public Safety in Austin. These numbers are used to track the types and amounts of controlled substances seized throughout the state. They are also used to justify positions and expenditures for the crime laboratory.

PROCEDURE

The monthly inventory sheet is used to compile the data for controlled substances received by each analyst. The inventory of substances received each day is entered on the sheet by placing the amount received by each category. The following guidelines should be used:

- Those substances that are listed by weights (such as cocaine, marihuana, heroin, etc.) are entered using either grams, kilograms, or pounds; grams and kilograms on the left, pounds on the right.
- All tranquilizers, synthetic narcotics, LSD, codeine tablets, and barbiturates are listed by dosage unit (# of tablets or capsules) next to the appropriate category.
- Methadone and codeine liquids are listed by volume in milliliters. If cocaine, methamphetamine, or amphetamine are in liquid form, the amount is listed in milliliters.
- All designer drugs are grouped together and listed either by weight or dosage unit.
- PCP cigarettes are listed under # CIGS, and PCP liquids are listed by volume in milliliters.
- The analyst's initials, the month, and the year should be included at the top of the monthly inventory sheet before turning it in to the appropriate person.

19 REPORTING GUIDELINES

SCOPE

To establish standards for reporting the results from the analysis of controlled substances, dangerous drugs, clandestine laboratory chemicals and other substances examined by analysts at the HPD Crime Laboratory.

PROCEDURE

Reports are entered into the LIMS system based on the HPD incident number which is located at the top of the report along with the date that the report is finalized. If applicable, the laboratory number will be entered after **Reference**. The names of all suspects listed on the submission form will be entered after **Suspect(s)**. If the name is listed as “fnu lnu” (first name unknown, last name unknown), none, **juvenile**, or unknown list the suspect(s) as **unknown**.

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

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

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

REPORTING GUIDELINES FOR ANALYTICAL RESULTS

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

Reporting Results of Controlled Substances and Dangerous Drugs

- General Reporting Examples of Identification
 1. Report the identification of a controlled substance as it appears in the *Texas Controlled Substances Act*.
 2. Precede the name of all substances identified with the word “**Contains**”. Marijuana and peyote will not be preceded with “contains” unless they

contain other materials.

3. If more than one controlled substance is identified in a sample, report them all after “**Contains**”.

*Examples: Contains Amphetamine and Methamphetamine
 Contains Cocaine and Phencyclidine
 Contains Cocaine and Marihuana*

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

- Reporting Marihuana, Marihuana Seeds and Hashish

1. Report plant substance identified as marihuana as “**Marihuana**” (not “**contains marihuana**”) and report the weight in ounces or pounds.
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 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*

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.

4. For cases that consist of marihuana seeds only, they may be reported as **“Marihuana seeds”** and the weight in ounces. 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”**.
5. Report hashish and liquid extracts as **“Contains Tetrahydrocannabinol”** and the weight in grams.

- Reporting Peyote Samples

For plants visually identified as *peyote* and analyzed to confirm the presence of mescaline, report as **“Peyote”** with the weight in grams. If the plant material cannot be visually identified as *peyote* or it is a powdered sample, report as **“Contains Mescaline”** along with the weight in grams.

- Reporting Mushroom Samples

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.

- Reporting Opium Samples

1. Morphine, codeine and thebaine are the opium alkaloids that are controlled substances. Non-controlled alkaloids include papaverine, noscapine and narceine. Opium samples, including commercial preparations such as Paregoric, should be reported as **“Contains Opium”** only if there is no heroin present and morphine and codeine are detected in combination with at least one of the other alkaloids. Samples which contain heroin should be reported as **“Contains Heroin”**.
2. Alternatively, the results can be reported as **“Contains Codeine, Morphine and (at least one other major alkaloid)”** with a footnote stating: **“These are commonly detected constituents of opium.”**

- Reporting Derivatives of Barbituric Acid

There are a number of derivatives of barbituric acid that are listed by name in the law. 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 a derivative of barbituric acid”**.

- Reporting Derivatives of 2-aminopropanal

There are a number of derivatives of 2-aminopropanal that are listed by name in the law. In those cases, report the name of the compound identified (for example, **“Contains 3,4-methylenedioxy-N-methylcathinone”**). If the compound is not listed by name, such as 4-methylethcathinone, then it should be reported as **“Contains a derivative of 2-aminopropanal”**.

REPORTING WEIGHTS AND VOLUMES

- If a controlled substance is identified in a powdered sample, chunk substance, or tar substance report the weight of the sample.
- Report the weight of liquid samples if a controlled substance is identified. The volume may also be reported.
- If the contents are identified include the weight of controlled substance tablets and capsules 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 instead of approximate on the report when an exact count is not determined (see Tablets and Capsules – General in the Analysis Guidelines Section for additional information).
- If a dangerous drug is identified in a sample (tablets, capsules, liquids, etc.), then no weight is necessary on the report.
- Except for marihuana, report the net weight in grams if the sample ranges from 0.01 grams to 1,000 grams. Weights greater than or equal to 1,000 grams may be reported in grams or in kilograms. Weights less than 0.01 grams should be reported as **“Less than 0.01 grams”**. Residue amounts should be reported as **trace**.
- 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. Report marihuana samples weighing more than one pound in pounds to at least one decimal place. If a marihuana sample weighs less than 0.01 ounces, the analyst should report the weight as **“Less than 0.01 ounces”**.

- 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

Reporting Abuse Units

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

Miscellaneous

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

- 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*

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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

** Insufficeint sample for quantitation*

- 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

- 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

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

- 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

- If a substance has been subjected to preliminary pharmaceutical identification without analytical 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. The notation “Pharmaceutical identification only” may be added as in the following example:

*Example: Indication Amitriptyline – Dangerous drug **
*Indication Acetaminophen – Over the counter **

** Pharmaceutical identification only*

- In the situation where a confirmation 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.*

- For cases processed according to the Excess Quantity Cases Section where photographs and representative samples are retained, the following footnote will be added to the report:

The Houston Police Department Crime Laboratory has photographed, determined the total weight of the substances and has retained representative samples as prescribed under the provisions of chapter 481.160 of the Texas Controlled Substance Act. The excess quantities may be destroyed 28 days after separate notification unless the Laboratory receives notice from the District Attorney's office before that date. The Houston Police Crime Laboratory will retain sufficient documentation as to the ultimate disposition of the narcotic(s).

- Reporting guidelines for disposed, dismissed, destroy, trace/residue possession, and paraphernalia cases can be found in the Guidelines for Processing Non-Active Cases Section.

Footnotes

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

- *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.*
- *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.

- *Not more than 200 milligrams of codeine per 100 milliliters or 100 grams and includes one or more nonnarcotic active medicinal ingredients.*
- *The HPD Crime Laboratory is accredited by ASCLD / LAB and the Texas DPS.*

Other Suggested Footnotes (taken from DPS)

- *A Dangerous Drug (Prescription Drug)*
- *The sample was consumed during analysis.*
- *Weight includes the weight of the rolling paper.*
- *Substances commonly found in opium.*
- *An analogue of gamma-Hydroxybutyric Acid (gamma-Hydroxybutyrate).*
- *Specialized footnotes may be used with the approval of a supervisor.*

20 ABBREVIATIONS

SCOPE

To provide a list of useful abbreviations.

ABBREVIATIONS

~	Approximately
2 C-B	4-bromo-2,5-dimethoxyphenethylamine
2 C-E	4-ethyl-2,5-dimethoxyphenethylamine
2 C-I	4-iodo-2,5-dimethoxyphenethylamine
AB	Analytical Balance
A/B extr.....	Acid/Base extraction
ACLS	Amera-Chem Library Search
Amp/amph.....	Amphetamine
approx	Approximately
AR	Administrative review
AVC	Abusable Volatile Chemical
BB	Bulky Balance
1,4-BD.....	1,4-butanediol
BZP	Benzylpiperazine
c (with line above)	Containing and/or with
cb.....	Cardboard box
cig.....	Cigarette
cig stub	Cigarette Stub
coc	Cocaine
CPP	Chlorophenylpiperazine
ch.....	Chunk
cry	Crystalline
DBZP	1,4-Dibenzylpiperazine
DD.....	Dangerous Drug
DIB.....	Drug Identification Bible
DMS	Dimethylsulfone
D.O.....	Destroy
EDIA	Evidence Destroyed in Analysis
EMS	Evidence Management System
est	Estimate(d)
ee	evidence envelope

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Extr.....	Extracted or Extraction
FID	Flame Ionization Detector
FTIR.....	Fourier Transform Infrared (Spectrophotometry)
g.....	Grams
GBL.....	gamma-butyrolactone
GC.....	Gas chromatograph
GHB.....	gamma-hydroxybutyric acid(γ -hydroxybutyrate)
Ind.....	Indication
JIMS.....	Justice Information Management System
kg.....	Kilograms
lb.....	Pounds
L.....	Liters
LIMS.....	Laboratory Information Management System
liq.....	Liquid
LSD.....	Lysergic Acid Diethylamide
Mari.....	Marihuana
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
MDPV.....	3,4-Methylenedioxy pyrovalerone
MeOPP.....	Methoxyphenylpiperazine
meth.....	Methamphetamine
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
num.....	numerous
NVS.....	No Visible Sample
neg.....	Negative
OLO.....	HPD On-Line Offense Report Access System
oz/ <i>ozs</i>	Ounces
PCP.....	Phencyclidine
PDR.....	Physician's Desk Reference

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PHI	Pharmaceutical Identification
pkg.....	Package
pl	Plastic
pos	Positive
Prometh.....	Promethazine
PS	Plant Substance
R _f	Retention factor (TLC)
RT	Retention time
Rx.....	Prescription
S	Sealed
STD.....	Standard
sub	Substance
TB	Top-Loading Balance
TFMPP.....	1-(3-Trifluoromethylphenyl)piperazine
THC.....	Tetrahydrocannabinol
TIC	Total Ion Chromatogram
TLC.....	Thin layer chromatography
TR	Technical review
UV/VIS	Ultraviolet/Visible (Spectrophotometry)
wt.....	Weight
zip.....	Ziploc(k)

21 COUNTING OF ITEMS AND TESTS

SCOPE

To provide guidelines for the counting of items and tests associated with cases processed by the Controlled Substances section. This information is entered into the case management system to document the completion of cases and to assist with monitoring section productivity.

CONTROLLED SUBSTANCE SECTION WORKSHEET

This form (commonly referred to as the Weekly Worksheet) is available to assist the analyst with documenting items, tests, and identifications for cases processed during a specified time period, but is not required. The information entered into the case management system will be considered to be the official documentation of work performed.

ITEM COUNTING GUIDELINES

Generally, the number of items to be counted for each case is based on the number of discrete pieces of evidence received and labeled for identification purposes. The following are examples to use in determining the number of items to be counted for a case:

- If 100 ziplocks of powder are received, but only 10 are tested and the remaining 90 are retained, then the number of items to be counted is 11. The 90 retained ziplocks are considered as 1 item.
- If a single ziplock of chunk substance is received but 16 chunks are sampled for separate spot testing, then the number of items is 1 (here the container of chunk substance is considered a discrete piece of evidence not the individual chunks).
- If pharmaceutical identification is performed for a case without spot tests, then the number of items is based on the number of containers and the different tablet/capsule logos. For example, pharmaceutical identification is performed on 5 bottles of tablets each having the same tablet logo; therefore, the number of items to be counted is 5. In another case one bottle contains tablets with three different logos; therefore, the number of items to be counted is 3.
- For evidence which is noted as “no visible sample”, count items that would routinely have a residue such as spoons, syringes, matchboxes, etc. Extraneous items which are grouped together for documentation (placed in the same Examination Sheet

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column) such as pieces of paper, rolling papers, matches, etc. should be counted collectively as one item.

TEST COUNTING GUIDELINES

In general the number of tests to be documented for a case is simply the total number of tests performed in the analysis of the case. Add the number of spot tests run to the number of microscopic exams to the number of instrumental analyses, etc.

How to Count Tests:

Marihuana - Add the number of microscopic exams to the number of Duquenois spot tests and the number of Duquenois-Levine spot tests performed.

Spot Tests - One test for each spot test performed. For example, if 2000 tablets are received and 50 Ferricyanide and 50 Marquis spot tests are performed then the number of items is 50 and the number of tests performed is 100.

UV – One test for each substance identified by UV. UV with a shift counts as one test. If an item has two substances and a UV is obtained for each substance, then that counts as two tests (ex. codeine and acetaminophen). No acceptable match or a negative result counts as one test.

FTIR - One test for each substance identified by IR. No acceptable match counts as one test.

GC/FID – One test for each sample quantitated by GC/FID. Insufficient sample for quantitation counts as one test.

GC/MS - One test for each sample run on each instrument.

TLC - One test for each solvent system used and one test for each standard spotted.

Microcrystalline - One test for each type of microcrystal from each item.

PHI - Any pharmaceutical ID on an item (tablet, capsule, ampoule, etc.) from an accepted source (PDR, Logo Search, Mexican PDR, etc.) is counted as a test. Just writing down the markings from the item is **not** a test. An unsuccessful attempt at identification counts as one test and is documented on the exam sheet.

Pharmaceutical identification will be counted as one test per column on the

examination sheet no matter how many dosages exist. For example, if 500 tablets are listed in one column, only one pharmaceutical test may be counted.

MISCELLANEOUS COUNTING GUIDELINES

- For cases that are re-examined by another analyst the counting of items and tests should follow the above guidelines.
- If you have any questions about how to count the number of items, tests, or note identifications, then consult with the section Lab manager or designee.

22 RE-ANALYSIS OF CASES

SCOPE

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

RE-ANALYSIS FOR PURPOSES OF TESTIFYING IN COURT

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

- The Controlled Substances Section Manager or designee will assign the case to an analyst who will receive the evidence from Centralized Evidence Receiving.
- The new analyst will process the case following normal procedures for analysis and use a new Examination Sheet for documentation.
- **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 Crime Laboratory no longer employs the original analyst.”

RE-ANALYSIS FOR ON-GOING QUALITY REVIEW OR INVESTIGATION

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.

- **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 section staff..**
- **The evidence packaging with seals and the contents may be photographed, if directed or appropriate.**
- **The evidence will be inventoried and weighed.**

- The analyst will proceed with re-analysis of items, as determined by the original Examination Sheets. The work previously conducted will be duplicated as much as possible.
- The analyst will document the results as directed. This may be in the form of a Re-analysis Worksheet or a new report.

23 GUIDELINES FOR PROCESSING NON-ACTIVE CASES

SCOPE

To provide guidelines for identifying case status, as well as for processing the evidence in cases which are identified as Non-Active, or for which a clear status is not available.

IDENTIFYING CASE STATUS

- Case status can usually be identified through JIMS and/or OLO as Active or Non-Active.
 - (1) Active cases are ones which have pending court dates, priority investigations, or have been requested by the grand jury or courts for one or more defendants. In addition, cases which are identified as bond forfeiture or to be warrant cases are considered Active. Information obtained from JIMS / OLO referencing this status should be included in the case file and labeled with the unique case identifier and the initials of personnel researching the status.
 - (2) Other cases may be identified as disposed (court accepted plea) or dismissed (charges dismissed by the court) **and are considered to be Non-Active**. Information for these cases obtained from JIMS / OLO should be included in the case file to reference the following: HPD Incident #, Cause #, Defendant, Charges, and Status. Printouts from JIMS / OLO should be labeled with the unique case identifier and the initials of personnel researching the status.
- The status of a case may not be readily available by reviewing JIMS or OLO or it may be determined that no drug charges have been filed for a particular case. Examples include Juvenile, Investigation Narcotics, Homicide, or cases filed outside of Harris County. Available information regarding the case status should still be included in the case file and labeled with the unique case identifier and the initials of personnel researching the status.
- In addition to the above situations, cases involving evidence tagged for Destruction, as well as evidence associated with Class C Misdemeanor Paraphernalia charges or Disposed Trace Cases, may require special handling or reporting procedures.

PROCESSING OF ACTIVE CASES

- If a case is found to be active for all listed defendants, then it should be processed according to the Analysis Guidelines Section and the Reporting Guidelines Section.

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- If a case has multiple defendants with a combination of status (Active / DISP / DISM), then the Active evidence should be processed and the remaining evidence may be retained. The JIMS status printouts should be included in the case file for the DISP / DISM evidence to support the decision to retain those items.

PROCESSING OF DISPOSED CASES

- If a case is identified as Disposed or Dismissed for all listed defendants, then only the items associated with the Disposed evidence need to be processed. Items associated with the Dismissed evidence may be retained.
- For items associated with charges that have been disposed, at least one positive test is required to report that a substance is indicated. At least two positive tests, one of which must be either FTIR or GC/MS, are required to report that a substance is identified. For plant substance, positive microscopic and chemical screening is sufficient for the identification of marijuana.
- A weight should be recorded for all items analyzed with the exception of items which are or which contain a residue (these are to be noted as "trace").
- For items analyzed, the report should contain a description of the evidence, the weight, and the results of analysis. If the analysis performed indicates the presence of a controlled substance, the report will reflect "**Indication [substance]**". If sufficient analysis has been performed to identify the presence of a controlled substance, then the report will reflect "**Contains [substance]**" or in the case of marijuana the report will reflect simply "**Marijuana**". If a dangerous drug is indicated or identified, then the report will include the notation that the substance is a dangerous drug. For items where a substance is not indicated or identified as a controlled substance or dangerous drug, the report will reflect "**No controlled substance identified**" (see No Controlled Substance Identification in the Analysis Guidelines Section).
- A technical and administrative review will be performed as for Active cases.
- An accreditation footnote should be included in the report as usual.

PROCESSING OF CASES WITHOUT A CLEAR STATUS

- Cases for which the status is not readily available by reviewing JIMS or OLO or for which no drug charges have been filed may be analyzed following the same analytical and reporting procedures as for Disposed Cases (see above).

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- A weight should be recorded for all items analyzed with the exception of items which are or which contain a residue (these are to be noted as "trace").
- A technical and administrative review will be performed as for Active cases.
- An accreditation footnote should be included in the report as usual.
- If additional information regarding the case status becomes available at a later date or if a request for a complete analysis is received, the case is to be handled per the instructions of the Section Manager or designee.

PROCESSING OF DESTROY CASES

- Some cases are received and tagged as "DESTROY" by the submitting officer. These are usually cases where there are no charges being filed and the offense is listed as "found property", "investigation narcotics", etc. There may be a notation such as "ADA Smith refused charges".
- Most of these cases will be filed in CER with no analysis. In the event that a destroy case is distributed for analysis (usually "investigation narcotics" charges) the same analytical and reporting procedures should be followed as for Processing of Disposed Cases.
- A weight should be recorded for all items analyzed with the exception of items which are or which contain a residue (these are to be noted as "trace"). If the gross weight of an item including packaging is used, this should be noted on the Examination Sheet and on the report.
- A technical and administrative review will be performed as for Active cases.
- An accreditation footnote should be included in the report as usual.

PROCESSING OF DISMISSED CASES

- For cases where all of the evidence has been identified as Dismissed for all defendants, no analysis is necessary. A report should be generated noting that no analysis has been performed due to the dismissal of charges per JIMS.
- The report should also include the associated Cause # for each listed defendant.

- The report will be reviewed to verify the information in the report is correct and that any documentation supporting the dismissal of charges is included in the case file.

PROCESSING OF PARAPHERNALIA CASES

- For cases that are identified as a class C misdemeanor paraphernalia charge, no analysis is necessary. A report should be generated noting that no analysis has been performed and it should include the submitting officer's description of the evidence as well as the following information:

“THE CASE WAS STATED AS BEING FILED AS A CLASS C PARAPHERNALIA CHARGE IN ACCORDANCE WITH HPD CIRCULAR NO. 09-1210-226. THE EVIDENCE IN THIS CASE WILL BE RETAINED BY THE LABORATORY FOR SIX MONTHS FROM THE DATE OF THE OFFENSE, AFTER WHICH TIME THE EVIDENCE WILL BE SUBJECT TO DESTRUCTION BY THE LABORATORY.

IF ANALYSIS IS REQUIRED, CONTACT THE LABORATORY AT 713-308-2600 AS SOON AS POSSIBLE.”

- The report will be reviewed to verify the information in the report is correct and that any documentation supporting the charge is included in the case file.

PROCESSING OF DISPOSED TRACE POSSESSION CASES

- For cases where the charge is identified as possession of a penalty group 1 or 2 controlled substance in an amount less than 0.01 grams and the case has been disposed, no analysis is necessary. A report should be generated noting that no analysis has been performed and it should include the submitting officer's description of the evidence as well as the following information:

“THE CHARGE(S) INVOLVE(S) THE POSSESSION OF A PENALTY GROUP 1 OR 2 CONTROLLED SUBSTANCE IN AN AMOUNT LESS THAN 0.01 GRAMS. THE CHARGES WERE DISPOSED ON XX-XX-XX PER JIMS. THE EVIDENCE IN THIS CASE WILL BE RETAINED BY THE LABORATORY FOR TWO YEARS FROM THE DATE OF DISPOSITION, AFTER WHICH TIME THE EVIDENCE WILL BE SUBJECT TO DESTRUCTION BY THE LABORATORY.”

- The report will be reviewed to verify the information in the report is correct and that any documentation supporting the disposition of charges is included in the case file.

Any questions regarding procedures for handling, analysis, or reporting of results for Non-Active cases should be directed to the Section Manager or designee.

End of Document

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