

*Approved by Director: Dr. Guy Vallaro***A. PURPOSE:**

Evidence submitted to the Unit can take various forms ranging from, powder materials, rocks, liquids, foods to paraphernalia. Analysts can plan analysis based on the type of matrix of the sample.

Analysts use their knowledge, training and experience to perform sample selection. Sample selection allows the analysts to form an opinion of what is contained in the items tested, an inference of the whole population cannot be made.

B. RESPONSIBILITY:

Analysts assigned to the Controlled Substance section.

C. DEFINITIONS:

1. "Like Items": evidence submitted where the appearance of the item leads one to believe that they are the same.
 - a. Example: zip lock bags all the same color all the same size and all containing a similar looking substance.
 - b. Example: clandestine tablets all the same shape, color and with the same imprint.
2. CHEP: Cyproheptadine dissolved in methanol or ethyl acetate (or in another solvent as appropriate) used as an internal check solution for GC/MS.
3. Sample Selection: selecting items to test or portions of items to test based on knowledge training and experience. Results apply only to the items tested, and relate to the whole population.

D. SAFETY:

Evidence submitted to the Unit can be from a wide range of sources including but not limited to Clandestine Laboratories, Body searches, Prisons and any variety of crime scene. Due to this all evidence must be handled using safety precautions and PPE as appropriate to the case materials. At a minimum, such PPE would include lab coats and gloves.

E. PROCEDURE:

Analysts using their experience, training and knowledge will make an initial assessment of a case based on the nature of the evidence submitted. This assessment will facilitate a determination of the most appropriate analysis to perform to identify the unknown. Chart CS-4:1 Controlled Substance: Evidence Matrices outlines the tests and extractions used per matrix type.

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- a. To report a controlled substance, a minimum of two confirmatory tests must be performed and provide consistent results as to the identity of the substance.
 - i. GC/MS and FTIR are both confirmatory tests. Most substances will be identified using 2 separate samples run by either GC/MS or FTIR. In general only GC/MS is most commonly used, with 2 portions or 2 like items being analyzed, but FTIR can be used at the analyst's discretion.
 - ii. Manufactured tablets with an identifiable imprint will be analyzed by searching for drug information using the logo and reputable literature, whether hardcopy or online. Information obtained will be documented in the casefile along with references. Such evidence may additionally be analyzed by other techniques (e.g., FTIR, GC/MS) for confirmation purposes, if necessary. Evidentiary tablet analyses can be stopped after information is obtained through a logo search only if tablets are not suspected to be illicitly produced and upon approval from either the Lead Examiner or Deputy Director. Subsequent reports shall convey the appropriate limitations to a logo-only search.
- b. If reporting a non-controlled substance, it is desirable to have two confirmatory tests in agreement. Due to the non-homogenous nature of street drugs samples; it is acceptable for analysts to report the presents of a non-controlled cut in a single portion of a sample. Controlled substances must be found in 2 portions of an unknown or in 2 like items on a case.
 - i. Cuts need not be identified unless they are the only substance found in the unknown, but may be reported as "Indicated based on a Mass Spectral Match only".
- c. Substances identified by GC/MS will be confirmed by analysis of a validated standard. This is for both controlled and non-controlled substances.

2. Sampling: sample selection is used (see C.3 above).

- a. For each set of "like items" within an evidence bag 2 randomly chosen items will be sampled and the analysis will proceed following the guidelines in CS-4:1.
 - i. Items chosen do not require homogenization since CT drug laws require only the presence of the drug be demonstrated. (From Connecticut Comprehensive Drug Law "Any material , compound, mixture, or preparation which contains any quantity of the following substance....)
 - ii. Laboratory reports will show that only 2 items of the group were analyzed and results obtained only represent the 2 items not the group as a whole.
 - iii. In cases where the set of like items is only 3 items the analyst can choose to analyze all three items; the report will show this.
 - iv. In cases where the two chosen items are analyzed and determine to be different more samples may need to be analyzed, the Lead Examiner or Deputy Director must be consulted. The plan determined must be documented in the case file.

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(a) Since the cases submitted to CS vary widely it is impractical to say that a specific number will be analyzed in the case of 2 varying samples. In general the following will be used as a guideline, knowing this may change depending on the specifics of the case involved:

(i) If the set is 10 or less items all the items will be sampled and analyzed.

(ii) If the set is 1000 or less items 10 additional items will be sampled and analyzed.

b. For single items two portions of the item will be sampled. (A single portion placed into each of two labeled test tubes.)

i. For approximate amounts to be taken by matrix type see CS-4:2.

3. Extractions/Sample Prep for GC/MS Analysis:

a. Direct: Many powders, rocks and clandestine, non-manufactured tablets or tablets with unidentifiable imprints are dissolved directly into CHEP in a labeled GC/MS vial.

b. 1:1 Petroleum Ether/Methanol: plant material is soaked in ~0.5ml of this solvent for a minimum of 30seconds (no maximum time) ~1-2 drops of the solvent is drawn off and placed into a labeled GC/MS vial for analysis.

c. Methanol or other appropriate solvent: (used to rinse paraphernalia) ~0.5-1mls of methanol is rinsed over the item using a transfer pipette. ~1-2 drops of the solvent is placed in a labeled GC/MS vial.

d. Borate buffer extraction: (used to clean some manufactured tablets, and to concentrate weak substances). Tablets, Capsules, and Powder/Rock-like substances run and found to be negative should be run using a borate buffer extraction. If not done in these instances the analyst will notate why this was not done.

~20-50mg (may be more depending on the nature of the sample) is placed in a test tube. ~1ml of saturated Borate Buffer is added this is allowed to sit for a moment and then an approximately equal amount of extraction solvent is added (chloroform or ethyl ether are generally used). The solvent layer is decanted into a labeled test tube using a glass Pasteur pipette, a portion of which is placed in a labeled GC/MS vial for analysis (the amount will depend on the nature of the sample).

e. Methanol /Hexane: (used to clean samples containing oils)

Place 3-5 drops of the liquid or approximately 0.5grams of sample (if food stuff) into a labeled test tube. Add approximately 2mls of hexane, and approximately 2mls of hexane-saturated methanol, vortex 4-5 seconds. Allow layers to separate. Carefully remove the lower, methanol layer using a glass transfer pipette and transfer this material to a labeled GC/MS vial.

f. Ethanol or other appropriate solvent: (used to rinse paraphernalia)

Approved by Director: Dr. Guy Vallaro

~0.5-1mls of solvent is rinsed over the item using a transfer pipette. ~1-2 drops of the solvent is placed in a labeled GC/MS vial.

- g. Other appropriate solvent: these are rare instances where the above listed solvents are not the best choice for the drug of interest. In these cases the analyst may choose to consult Clark's or another reference to determine a suitable solvent for the suspected analyte. The solvent used will be noted on the evidence summary sheet or in the case notes.

4. Controls:

Positive and negative controls are used to demonstrate that both the method works in extracting a drug from the matrix it is in and that the laboratory supplies/methods have not introduced any substances to the unknown. A positive and negative control is run with each "batch" of samples, not for each sample. (Example: If plant material is sampled from each of 4 cases one positive and one negative control can be prepared as long as the extraction process occurs at the same time.)

Positive and Negative controls are to be prepared and handled as close to case samples as is possible. If samples need to be concentrated or diluted the same must be done to the negative controls. If samples need to be run in an additional temperature program on the GC/MS the negative controls must also be rerun. The positive control need not be repeated if the ability of the procedure to extract the substance of interest has already been demonstrated in the initial run. The negative is repeated to assure there is not a contaminant introduced during the additional steps.

- a. Negative Control: A "Negative control" is prepared in conjunction with the sample for each type of extraction. Negative controls will be prepared in a manner as close to the method as possible, (note it is understood that the sample matrix cannot always be duplicated). Any reagents, solvents or disposable glassware (etc.) used in the sample preparation will be used to create a Negative control.
- b. Positive Controls: Positive controls will be run as appropriate for the extraction method being performed. Controls can be prepared by the Lead Examiner and analysts can be provided with "stock" controls by matrix. Positive controls are prepared for the major types of sample matrixes:
 - i. Plant material: tea - caffeine is the component that is identified
 - ii. Powder/rock: a powder standard preferably a non-controlled substance
 - iii. Tablet: an over the counter tablet such as diphenhydramine tablet
 - iv. Residue: Test tubes will be prepared with dried known components
 - v. Liquids:
 - (a) Aqueous or oil based: non-controlled substances or controlled substances in concentrations of 1mg/ml or less are used.

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(b) "Drinks" (fruit drinks, alcoholic substances, coffee, milk etc.): a reasonable attempt will be made to obtain a "blank" control that is a matrix that mimics the submitted sample. Example if a coffee is submitted, coffee will be obtained. If a red fruit juice is submitted a red fruit drink will be purchased. The "blank" material will be spiked with a non-controlled substance or controlled substance in a concentration of 1mg/ml or less in liquid form.

(c) Diversion Cases: Drug Control generally submits exemplars with each diversion case; if an exemplar is not provided consult the Lead Examiner or Deputy Director to determine if one must be obtained or if another control can be prepared.

vi. Food Stuff: a reasonable effort will be made to obtain a material that is a similar matrix to the substance submitted. Example: Brownie submitted for analysis a brownie or cake like substance will be obtained. The material will be spiked with an appropriate validated reference material. This can be a non-controlled substance or a controlled substance in a concentration of 1mg/ml or less in liquid form. A notation will be made of the substance used as the control material.

vii. Other matrices - the analyst should consult the Lead Examiner. Attempts will be made to keep the control similar to the sample matrix. A notation will be made of the substance used as the control material.

5. Photographs:

- a. Photographs are taken of all case materials as a record of the physical appearance of the evidence and as a way to demonstrate that multiple items "appear" alike.
 - i. **Note:** in large cases the photographs need not show all the items individually, as to demonstrate the number of items (i.e. such as with a case containing hundreds of glassine bags, the bags can be piled together to show they are alike, they need not be laid out to show the number of items).
- b. Memory cards/CDs or other appropriate data storage device containing the original digital image are maintained in the Unit until backed-up.
- c. Copies of the photographs are printed and placed in the case file.

6. Weight:

- a. Evidence in the form of Powders, Rocks and Plant materials are all weighed. Analysts must consider state and federal criteria weight limits when determining how to proceed with the weighing of a case; is direct weight required or is weight with packaging sufficient? When reporting weights uncertainty of measurement must be considered. (See SOP CS-5).
- b. For multiple item submissions the weight of each group of like items is reported along with the weight of the items analyzed.

Approved by Director: Dr. Guy Vallaro

- i. If the weight of a group of items is below State or Federal criteria weights, the gross weight is taken and reported. Similarly, the individual item weights can be reported as gross weights. (The analyst may report a direct weight of individual items, based on case specific circumstances.)
- ii. If the weight of the group of like items is above state criteria levels then the net weight of the group of items is reported, similarly the weight of the individual items analyzed will be reported as a direct weight (without packaging).

7. Solubility:

- a. This is performed on potential cocaine samples when the form of the drug is to be reported. Substances that are insoluble in water are likely to be the free base form of the drug, samples that are soluble in water are likely to be the salt form of a drug.
- b. Place a small amount of the unknown in a clean test tube add DI water and observe.
 - i. If the substance dissolves it is likely the salt form of the drug. Two portions of a single item or two items from a set of samples must be tested in this manner to be identified as the salt form of the drug. The sample may also be run in FTIR to determine salt form.
 - ii. If the substance does not dissolve it is likely the free base form of the drug. In this case one portion (or one item) is tested in this manner and one portion (or item) is tested by FTIR. Agreement between both tests allows the substance to be identified as the free base form of the drug.
- c. If the substance is partially insoluble or questionable two portions (or two items) are tested by FTIR to determine the form of the drug. Note: since the form of the drug is only reported for cocaine cases, the analyst can choose to analyze evidence using only FTIR. This will confirm the presence of cocaine and the form.

8. MiScope®:

- a. This is used to identify cystolithic hairs on marihuana. Cystolithic hairs are present on marihuana and are helpful in distinguishing marihuana plant material from non-marihuana plant material.
- b. The identification of cystolithic hairs or the absence of these hairs is a screening tool it is not a confirmatory tool.
- c. The MiScope® is a combination microscope digital camera. The magnification of this device is 40-140X. The device is plugged directly into a computer allowing digital pictures to be taken.
- d. Using the MiScope®:
 - i. Place a small amount of plant material on a clean piece of paper

Approved by Director: Dr. Guy Vallaro

ii. Place the MiScope[®] over the material use the zoom feature to magnify the area of interest and press the capture button to take the picture.

iii. Print the picture: include the case number, item number, date, and conclusion on the picture (handwritten or computer generated).

e. The presence of cystolithic hairs demonstrates that the plant material is likely marihuana.

f. If cystolithic hairs are absent it is likely that the plant material is not marihuana.

9. GC/MS

a. Gas Chromatography Mass Spectrometry is a confirmatory test. (See Sop CS-7)

b. The evidence analyzed in the laboratory can be analyzed by GC/MS or FTIR.

10. FTIR

a. Fourier Transform Infrared Spectrophotometer is a confirmatory test. (See SOP CS-8)

b. This instrument is can be used by the Unit to distinguish the salt and free base forms of Cocaine if required. Samples which are insoluble in water or partially soluble in water and are identified by GC/MS as cocaine are run by FTIR to determine if the cocaine is of the free base form. It is also used as a confirmatory test, where appropriate.

c. The FTIR can also used for the identification of evidence items and supplement GC/MS data.

F. **REFERENCES:**

1. Clark's Isolation and Identification of Drugs in Pharmaceuticals, Body Fluids, and Post-Mortem Materials, The Pharmaceutical Society of Great Britain.
2. Connecticut Comprehensive Drug Laws.
(http://www.ct.gov/dcp/lib/dcp/pdf/drug_control_pdf/drug_laws_september_2010_for_web_december_revision.pdf)
3. State of Connecticut Department of Consumer Protection Controlled Drug Schedules, Violations & Penalties: http://www.ct.gov/dcp/lib/dcp/pdf/drug_control_pdf/2010_cs_violation.pdf
4. Federal Trafficking Penalties: <http://www.justice.gov/dea/agency/penalties.htm>.

G. **REFERENCES FOR TABLET IMPRINT CODES** (not-inclusive):

1. The Drug Identification Bible
2. Physicians' Desk Reference
3. Web sites such as Drugs.com, RX list, manufacturer's web sites

CS 4 Sample Matrices

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Page 8 of 8

Rev#	History
4	In section 4, removed language “maybe reagent Blank” in or order to be more consistent with our procedures. General formatting changes throughout document. Accepted the changes and removed the red underlined edits.
5	Replaced laboratory and section with Unit. Made minor grammatical improvements. Updated tablet analysis to mimic GC/MS SOP.

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