CS 4 Sample Matrices Document ID: 1295

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A. PURPOSE:

Evidence submitted to the Unit can take various forms ranging from powder materials, rock-like material, 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 Unit.

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.

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 provides guidelines for the tests and extractions that may be used per matrix type.

1. General Information:

- 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 two separate samples run by either GC/MS or FTIR. In general, only GC/MS is most commonly

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used, with two portions or two like-items being analyzed, but FTIR can be used at the analyst's discretion.

- ii. Due to the non-homogenous nature of street drugs samples; it is acceptable for analysts to report the presence of a non-controlled cutting agent in a single portion of a sample.
- iii. Cutting agents need not be identified unless they are the only substance found in the unknown.
- b. Substances identified by GC/MS will be confirmed by analysis of a validated reference 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, two 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.
 - ii. Laboratory reports will show that only two items of the group were analyzed and results obtained only represent the two items not the group as a whole.
 - iii. In cases where the set of like items is only three 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 determined to be different more samples may need to be analyzed, the Lead Examiner or higher must be consulted. The plan determined must be documented in the case file.
 - 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. Procedure Controls:

Positive and negative procedure 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 four 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 control. If samples need to be run in an additional temperature program on the GC/MS, the negative control 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 control is repeated to assure there is not a contaminant introduced during the additional steps.

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- 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. 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 1 mg/ml or less are used.
- (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 higher 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.

4. Weight:

- a. 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.

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5. MiScope®:

a. This is used to identify cystolithic hairs on cannabis. Cystolithic hairs are present on cannabis and are helpful in distinguishing cannabis plant material from non-cannabis 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
- 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. Include the case number, item number, date, and conclusion in the picture (handwritten or computer generated). This image can be printed with the case file or stored electronically within LIMS.
- e. The presence of cystolithic hairs demonstrates that the plant material is likely cannabis.
- f. If cystolithic hairs are absent, it is likely that the plant material is not cannabis.

6. GC/MS

- a. Gas Chromatography / Mass Spectrometry is a confirmatory test. (See SOP CS-7)
- b. Sample preparation procedures are outlined in CS-7.

7. FTIR

- a. Fourier Transform Infrared Spectrophotometer is a confirmatory test. (See SOP CS-8)
- b. This instrument can be used by the Unit to distinguish the salt and free base forms of Cocaine if required. It is also used as a confirmatory test, where appropriate.
- c. The FTIR can also be used for the identification of evidence items and supplement GC/MS data.

F. **REFERENCES**:

- 1. Clarke'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. (LawBook.pdf (ct.gov))
- 3. Federal Trafficking Penalties: http://www.justice.gov/dea/agency/penalties.htm.
- 4. The Drug Identification Bible
- 5. Physicians' Desk Reference