ABOUT THIS DOCUMENT

Procedures contained in this document are reviewed annually. Changes, if any, are acknowledged by staff. Obsolete / retired versions are archived and retained in the laboratory for at least two years.

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A. EVIDENCE CONTROL

1. Chain-Of-Custody

At the time of receipt, each case will receive a laboratory identification number. Whenever possible, all pertinent information contained on the Receipt/Contract for Examination form will be completed with every submission to the HETL Forensic Chemistry Section (FCS). The receipt will be completed in triplicate and the copies distributed as follows:

a. White copy (original) to submitter or case file
b. Yellow copy to case file
c. Pink copy placed in the appropriate file located in the evidence technician office.

The sealed package weight (SPW) will be taken and recorded on the evidence form, and package sticker, along with a description of the evidence received. The *sealed package weight (SPW) is the weight including the envelope, box, etc. containing the evidence.*

The recipient will mark all evidence and corresponding paperwork for identification with the laboratory number**.** Evidence will be secured in either the evidence safe, evidence refrigerator, evidence freezers, evidence cabinet in the laboratory, or evidence room. Upon relinquishing control of evidence, transfers will be noted on the reverse side of the pink Receipt/Contract for Examination form. The back of the pink copy serves as the internal chain of custody document.

When placing evidence into Evidence Storage, the reverse side of the pink copy will be completed. Upon completion of the casefile the pink sheet will be placed in the accountability file located in the evidence technician office. This copy must be annotated each time someone removes and returns evidence from/to evidence storage. This includes intra-laboratory transfers. Once a case is completed, all pink sheets are maintained in the accountability file until such time as the evidence is returned or destroyed.

Evidence to be analyzed will be removed from evidence storage and brought directly into the laboratory for processing. A description of the item(s) and the condition of the packaging will be noted in the analyst's case notes.

Whenever possible, care should be taken to not cut, or break original evidence seals. However, it is recognized this is not always possible depending on packaging and placement of seals. During analysis the unsealed evidence will be under the control of the analyst. If the analyst must leave his/her work area for an extended period, all evidence will be placed in the analyst's lockbox or evidence cabinet or secured in some fashion. Samples, or aliquots, of the evidence will be taken for analysis. The samples/aliquots will not be tracked on the chain of custody during the testing process.

After sampling, the evidence will be returned to the original package, if possible, and sealed. Take care not to seal over any bar codes from the submitting agency to prevent issues with scanning upon return. Staples should be avoided to prevent piercing the heat sealed bags within the external envelope. If new packaging is required, which differs from the originally submitted exterior container, this change shall be noted on the chain of custody and analyst's worksheet. If items are separated or sub-items are created, these shall be documented on the analyst's worksheet and tracked on the chain of custody. The seal will be dated, signed, or initialed by the analyst and the post sealed package weight (pspw) recorded on the container and in the case notes. Processed evidence will be placed into evidence storage until final disposition. The reverse side of the pink receipt form will be completed and retained in the appropriate accountability file.

Upon final disposition (return to submitting agency or destruction), the Receipt/Contract for Examination Form will be removed from the accountability file and placed in the case folder.

B. Evidence Handling and Packaging

Preserving the integrity of evidence is crucial for proper interpretation and future admissibility at trial. Integrity of evidence is maintained through two practices:

1. Proper Handling

All laboratory personnel will handle submitted materials in a manner that assures the integrity of the evidence. Prior to initiating and during the processing of evidence, the analyst will employ the following practices:

- The work area will be clean and free of excess debris –
    - Countertops are cleaned when dirty, or as needed by the Analyst
    - Trash is removed daily, or when necessary
- All glassware, tools, spatula's, etc used in conjunction with examining evidence will be clean.
- Test tubes, capillary pipettes and Pasteur pipettes are used only once, then discarded.
- To prevent cross contamination of samples, only one case will be opened and sampled by the analyst at a time. Additionally, in cases involving multiple submissions, only one item will be opened by the analyst at a time. This does not mean that multiple cases cannot be 'batched' for analysis, but rather, only 1 item of evidence shall be open at a time for sampling, manipulation, etc.
- All evidence will be stored under proper seal (see below).

- Reagents and solvents will be kept in closed containers, and labeled with identity, lot number, who prepared (if applicable), and any special storage requirements.

2. Proper Packaging

All evidence must be packaged in a manner that ensures its integrity. Therefore, all evidence must be retained under proper seal. **Proper seal** is defined as: containers sealed to prevent the loss of contents and secured in a manner such that entering the container results in obvious damage or alterations to the container's seal. The seal must also have initials across it, at a minimum. Evidence that is actively being examined by an analyst need not always be sealed, but shall be secured when not actively being sampled, analyzed, etc.

After the exhibit has been sampled for analysis, the exhibit will be re-packaged. The analyst will seal the container with evidence tape, place their initials and date across the tape seal.

II. QUALITY ASSURANCE

A. Equipment Maintenance and Calibration

Refer to Appendix A, Appendix B, and the Quality Manual.

B. Reagents, Standards, and Quality Control Materials

Standards purchased from an approved vendor shall be used for comparison for identifications and confirmations. Refer to SOP Manual and Quality Manual for standard use and handling requirements. DEA non-exempt controlled substance standards have special inventory and destruction requirements. It is the analyst's responsibility to ensure proper handling and storage of all standards.

The SDD GC/MS process for standard evaluation and approval is as follows:

- All compounds should be named using the following convention
  [COMPOUND NAME] [LOT NUMBER] Exp Date Run Date

- New Compounds
  - A Certificate of Analysis shall be obtained from the vendor specific to that lot number.
  - **Reference Spectra**: The standard shall be run on each appropriate analysis method and instrument, used to update the reference spectra and retention time, and added to the Quant QEdit list. A print off from each analysis method and instrument shall be provided to the Quality Manager for review.
  - **Library Entry**: The standard shall be compared to an approved external GC/MS library and achieve a library match of 80 or greater, prior to the standard being added to the HETL library. If an acceptable library match is achieved the standard shall be added to the HETL library using the *Compound Name, Lot Number and Expiration Date* as the entry name. A print off of the external library match shall be provided to the Quality Manager for review.
  - The Quality Manager shall review each packet to ensure the lot number, expiration date, retention time, library comparison and library entry are acceptable. The standard will be entered into the tracking excel sheet and a PDF of the packet will be saved on the K drive for easy reference.
- New Lot Number of Existing Compounds
  - A Certificate of Analysis shall be obtained from the vendor specific to that lot number.
  - **Reference Spectra**: The standard shall be run on each appropriate analysis method and instrument, used to update the reference spectra and retention time, and updated on the Quant QEdit list. A print off of at least one analysis method from both instruments shall be provided to the Quality Manager for review.

- **Library Entry**: The standard shall be compared to an approved external GC/MS library and achieve a library match of 80 or greater, prior to the standard being added to the HETL library using the compound name, lot number and expiration date as the entry name. If an acceptable library match is achieved the standard shall be added to the HETL library. A print off of the external library match shall be provided to the Quality Manager for review.
- The Quality Manager shall review each packet to ensure the lot number, expiration date, retention time, library comparison and library entry are acceptable. The standard will be entered into the tracking excel sheet and a PDF of the packet will be saved on the K drive for easy reference.

The SDD FTIR process for standard evaluation and approval is as follows:

- New Compounds and New Lot Number of Existing Compounds
  - A Certificate of Analysis shall be obtained from the vendor specific to that lot number.
  - The standard shall be run on the appropriate analysis method, compared to an approved external FTIR library, and achieve a library match of 80 or greater, prior to the standard being added to the HETL library. If an acceptable library match is achieved the standard shall be added to the HETL library using the *Compound Name, Lot Number and Expiration Date* as the entry name. A print off of the standard spectra and external library match shall be provided to the Quality Manager for review.
  - The Quality Manager shall review each packet to ensure the lot number, expiration date, library comparison and library entry are acceptable. The standard will be entered into the tracking excel sheet and a PDF of the packet will be saved on the K drive for easy reference.

Refer to specific analysis procedure, SDD Reagent Logs, and the Quality Manual for more information regarding reagents and quality control measures.

C. QUALITY CONTROL

Functional checks will be performed to check the performance of equipment and regents used (either at regular intervals or while testing samples). Control checks will be performed during the analysis or testing process. These checks are used to:

- Determine the performance of the analytical or testing system.
- Quantitate (if possible) the variability of results from the analysis or test in terms of precision and accuracy.

The data from the check analyses will be compared with the expected values. Any significant difference (as determined by the analyst) shall be reported to the Section Supervisor.

To determine the proper operation of the balances, weekly and daily checks will be performed using calibrated weight sets. The weight sets should be calibrated to at least a Class II requirement or higher. Results will be recorded on the balance check form. Weekly checks will be done using a range of weights that covers the applied use of the balance and daily checks will be done using one lower mass and one higher mass, as outlined on the balance check form. Checks shall only be required on balances used to report out weights on days/weeks when the balance is in use.

To determine the proper operation of the GC/MS and/or lack of solvent contamination, the analyst will run and evaluate a blank containing the Internal Standard and appropriate solvent, if applicable, between each case sample. The volumes of internal standard and solvents for the blank should be prepared as close to the sample preparation as possible.

To determine the lack of contamination during multisolvent (complex) extractions, one procedural extraction blank per extraction set will undergo each step of the extraction procedure, alongside the samples, prepared the same as the samples for GC/MS analysis, and run under the same GC/MS conditions as the sample.

Evaluation of blanks is used to determine if a sample needs to be re-run or if there is a need for instrument maintenance. A clean blank and procedure blank, when applicable, is required for confirmation of a controlled substance.

D. CRITERIA FOR EVALUATING BLANKS IN GC/MS ANALYSIS

An acceptable blank will be evaluated using the same instrument method as the sample, and contain no target compounds as listed on the Quant QEdit Report for the method used and/or other compounds, at the discretion of the analyst, having a signal to noise ratio of greater than 3:1. An internal standard peak must be present in the blank meeting all acceptability criteria outlined in section V-B-2 of this manual. It is noted that a minor secondary peak has historically been present in the DFTPP blank with a signal to noise ratio >3:1 containing ions 271 m/z, 290 m/z, 438 m/z, and 458 m/z. The analyst should evaluate this peak during analysis to ensure it is not a target compound, however no remedial action or additional documentation in the casefile is required if this secondary peak is present in a DFTPP blank at a signal to noise > 3:1. If a standard is updated for a sample, following the evaluation of the corresponding blank, the blank must be reprocessed, along with the sample, for proper evaluation.

In cases where the blank fails these criteria, the subsequent case sample vial will be re-run and/or re-extracted. The analyst will indicate on the printout that the sample is being rejected (or similar terminology), why the sample is rejected, the date of rejection, and the initials of the analyst. Failed blanks will be retained in the case file.

E. POLICY FOR REPORTING UNCERTAINTY OF MEASUREMENTS

When estimating the uncertainty of measurement, all uncertainty components which are of importance shall be taken into account using appropriate testing procedures. Refer to the SDD UoM Procedure for details.

Single weighing events: A single weighing event is defined as placing the empty weighing vessel onto the balance and obtaining a 'tare', and then adding the measurand (item to be weighted) to the vessel while it remains on the balance and recording the net weight. The uncertainty will be reported based upon the calculated total uncertainty specific to the balance used at 95.45%, K=2.

Multiple weighing events: Multiple weighing events are defined as placing the empty weighing vessel on the balance and obtaining a 'tare', removing the vessel from the balance, adding the measurand, and then returning the vessel with measurand to the balance to obtain the net weight. The uncertainty reported is the calculated total uncertainty specific to the balance used (standard uncertainty/U*2 or expanded uncertainty)(# of items being considered)(# of weighing events). The result is expressed at 95.45%, K=2.

Combining weight to report a total weight of multiple single weighing events or multiple events: When multiple weights are combined to represent a single weight, the uncertainty reported is the sum of the total expanded uncertainty for each weighing event, or the most conservative number of weighing events, or uncertainty estimate, is applied to all samples weighed for that item.

Additional information related to UofM in Solid Dose Drug Chemistry is contained in the "Solid Dose Drug UofM Procedures Document", on file with the Quality Manager and on SharePoint. Additionally, the Quality Manager has Certificates of Calibration for each balance used to report Solid Dose Drug net weights, repeatability data, and verified calculations related to final reporting values of UofM on each balance.

III. GENERAL CASE HANDLING PROCEDURES

A. GENERAL OBSERVATIONS

1. Package Condition

The analyst will note the condition of the evidence package: i.e.: Sealed with Initials, Sealed without Initials, or Unsealed. Initials should be present across the seal of the packaging. If initials are not present it shall be noted in the case folder.

2. Identification Numbers

The analyst will ensure all identification numbers agree with the Receipt/Contract for Examination form.

The analyst will document the HETL case number and, if applicable, the submitting agency's item number in the case notes.

3. Evidence Description/Discrepancies

When applicable, the analyst will ensure evidence description(s) reasonably agree with the description provided by the submitting agency. The purpose of identifying a discrepancy is to ensure the correct piece of evidence was submitted, the correct piece of evidence is being examined, and the evidence has not been compromised in any way prior to analysis.

If a **minor** discrepancy is found the analyst will continue with testing and the evidence description on the Certificate of Analysis will serve as customer notification of the discrepancy. Examples of a minor discrepancy may include:

- evidence was submitted listing 58 white tablets and the analyst counted 57 white tablets
- evidence was submitted as 8 tied bag corners of white powder and the analyst counted 10 tied bag corners of white powder
- evidence was submitted as 950 envelopes of tan powder and the analyst counted 943 envelopes of tan powder
- evidence was submitted as a bag of tan powder and the analyst found a bag of tan powder, a lighter, and an empty bag with residue

These examples demonstrate evidence that still reasonably agrees with the submitters description and no action is required by the analyst.

If a **major** discrepancy exists between evidence descriptions no testing will be conducted until reconciliation between the analyst and the submitter (or their representative) is accomplished. Examples of a major discrepancy may include:

- evidence was submitted listing 58 white tablets and the analyst counted 3 green tablets
- evidence was submitted as 8 tied bag corners of white powder and the analyst counted 1 bag of tan powder
- evidence was submitted as 950 envelopes of tan powder and the analyst counted 1500 envelopes of tan powder
- evidence was submitted as a bag of tan powder and the analyst found a bag of green plant material

NOTE: refer to Quality Manual regarding the procedures for addressing evidence discrepancies

The analyst will include a general description of the submitted evidence. The description will include the type of container and its contents. Abbreviated descriptions (i.e.: OSEE = One Sealed Evidence Envelope) may be used in the description. Abbreviated descriptions must be interpretable by other staff Chemists. A list of abbreviations is maintained on SharePoint. Common abbreviations such as g for gram are viewed as common knowledge, should be readily recognized by other qualified analysts, and need not be explicitly detailed in the abbreviations document. Chemical abbreviations are maintained in the HETL Chemical Hygiene Plan.

If the item is difficult to describe or a more detailed description is necessary, the analyst may document the item by sketching it in their notes, or by taking a picture using a state controlled camera (not a personal camera or cell phone). If a picture is taken, a ruler shall be included in the photo. The photo shall be printed for the case file and have the following items documented on the printout: HETL case number, item number(s), analyst initials, and date.

4. Sealed Package Weight

Sealed Package Weight = *the sealed package weight (SPW) is the weight including the envelope, box, etc. containing the evidence*.

The sealed package weight should be recorded in the case notes. Reconciliation action should be taken if, at the time of analysis, the evidence seals are not intact and/or the SPW of the sample differs significantly (>10%) from the weight at the time of submission.

If seals are not intact or initialed a note will be made on the worksheet by the analyst and a description of the issue (seals were never intact, seals were intact but are now compromised, or seals are present but not initialed). If seals were never intact (ex: heat sealed bag never sealed or adhesive cover was never removed from bags with built in seals) a note will be made on the worksheet and reported on the Certificate of Analysis. If seals were damaged or compromised no testing will be conducted until another

analyst reviews the packaging integrity and a reconciliation between the analyst and submitter is accomplished.

If the SPW differs significantly from the weight at the time of submission, no testing will be conducted until another analyst reviews packaging integrity or reconciliation between the analyst and the submitter (or their representative) is accomplished.

5. Gross Weight (if applicable)

Note: If the nature of the sample requires a net weight to be obtained, then (if possible) a gross weight will also be obtained.

Gross Weight = ***Gross weight will include any packaging directly touching the suspected drug.***

Prior to beginning analysis, a **gross weight** of the drug submission will be obtained and written in the case notes.

B. ANALYSIS

1. Net Weight

Net Weight = ***the net weight is the weight of the drug specimen that includes no packaging.***

The **net weight** of the drug sample will be obtained after removal of the drug from the package. The analyst should remove any extraneous debris from the sample prior to obtaining the net weight, where possible. If extraneous debris is not able to be removed, a note shall be made on the examination worksheet. This weight shall be recorded on the worksheet.

2. Reserve Weight

Reserve Weight = the weight of the drug specimen after sampling has occurred, but before it has been repackaged.

The reserve weight is to be taken by the analyst following sampling, but prior to repackaging. This weight shall be recorded on the worksheet.

3. Evidence Sampling Plan

Ideally, the evidence sampling requirements should be detailed at the time of submission or upon conference with the investigating officer or representative from the prosecutor's office (District Attorney/Attorney General). However, it is recognized that after analysis starts, and depending on the results, the sampling plan / approach may change. Changes in the sampling plan will be documented within the case notes, and if significant, may require notification to the customer before the final report is issued by the analyst.

When no specific sampling instructions have been provided by the client, the analyst may need to contact the customer to discuss the analysis and determine what type of sampling is needed. Alternatively, it may be reasonable for the analyst to use their experience after examining the items to determine the best path for analysis at the least cost for the customer.

Sampling decisions in cases involving multiple similar items, or dosage units, may need to be based on either the Administrative sampling plan, or a statistically valid method that is recommended/accepted by major peer institutions (such as SWGDRUG, DEA or ASTM) if conclusions regarding the entire population are needed. It is recognized that the type of sampling may also vary based on the type of case (i.e., criminal charge), the number of submitted items, the total weight(s), and the wishes of the customer.

When the customer requests deviations, additions, or exclusions from the sampling methods, it shall be recorded in the case record with appropriate sampling data and test results. The final report to the customer shall be clear as to what was sampled and what the results are, and if they are specific to items tested, or if statistical conclusions regarding the entire population are associated with the analysis. A method deviation comment will be communicated on the case report. Any email or record of phone conversations with the customer regarding sampling will also be retained in the case record/file.

HETL will employ a multifaceted approach to examining submitted drug evidence: Including provisions to maximize the resources of the laboratory and to reduce the cost to the customer. This multifaceted approach will include components that include drug item reduction sampling, administrative sampling, and 2 levels of hypergeometric sampling. Each of which is detailed below.

Drug Item Reduction Program: The Drug Item Reduction Program allows for the analysis of probative items within a case to maximize the resources of the laboratory and to minimize the cost to the customer.

In every case, the most significant items in terms of quantity and schedule are analyzed. This “rule of thumb” cannot address every drug case scenario. Consideration must be given to the information contained on the Request for Laboratory Examination. This includes things such as the specific charges or types of offense, items unique to a single suspect, the statement of fact and examinations requested, and the descriptions of evidence submitted as well as the chemist’s visual inspection of the items and experience after opening and viewing all contents and documents associated with the case.

If at a later date it becomes apparent that items not initially analyzed require analysis for successful prosecution, then upon re-submission and/or request, that item(s) will receive top priority at the laboratory.

Procedures related to Drug Item Reduction

Syringes should only be analyzed if it is the only item in the case and has been specifically requested by the customer.

Residues, cigarettes, or cigarette butts will not be analyzed when measurable quantities of the associated drugs are also included among the items submitted.

Pharmaceutical preparations should be visually examined using pharmaceutical identifiers and appropriate reference materials. If all items are visually consistent (one homogenous population), then 1 tablet will be confirmed. The report will indicate total number of tablets submitted, 1 tablet selected and found to contain 'xxx'.

If identical intact, marked pharmaceutical preparations (e.g., tablets or untampered capsules) are present in multiple items, full analysis is required for only one item. Those preparations not analyzed may be reported as "Not Analyzed" or "Visually consistent with…"

Partial pharmaceutical preparations need not be analyzed when intact pharmaceutical preparations or measurable quantities of the same drug(s) are present. Analysis of partial pharmaceuticals may be required if it is suspected that the partial pharmaceutical is of a higher penalty group than the other items.

Items not analyzed will be clearly documented in case notes, and where appropriate to understanding the results, may need to be included in the report sent to the customer.

Example 1: Submitted evidence includes a plastic bag corner containing tan powder and a 1cc syringe with 10 units of liquid. The tan powder would be analyzed, and the syringe would not.

Example 2: Submitted evidence includes five tablets containing oxycodone and a plastic straw section with residue. The tablets would be analyzed, and the straw section would not.

Example 3: Submitted evidence includes five tablets containing alprazolam and a plastic straw section with residue. The tablets would be analyzed, and the straw section would not unless information on the submission form indicates that the straw section was used for a different drug, or the residue is markedly different from the other material in the submission, or there is reason to believe that the residue from the straw is comprised of a drug in a higher penalty group.

Example 4: Submitted evidence includes a plastic bag of plant material and a glass tube smoking device with white residue. Both the plant material and the smoking device would be analyzed based on different penalty groups.

Administrative Sampling: The administrative sampling plan will be used in cases to answer a specific legal question(s), or when items that appear to be similar, involve drugs with weight thresholds. If more specimens than listed in the hypergeometric sampling plans need to be analyzed to meet the weight threshold, the customer may need to be contacted for consultation regarding the best approach to sampling.

Possession-Furnishing-Trafficking with/without weight thresholds:

If multiple items (or sub-items) are present in the case, and analysis of additional items will meet a weight threshold, then sufficient items to meet the threshold will be fully examined (confirmed).

If a weight threshold cannot be met by examining additional items, then the analyst shall select the largest item (sub-item) and confirm. All other items may be reported as untested, or the analyst may weigh the other items, report their weight on the report, but clearly indicating the confirmation (and associated weight) is specific to the item confirmed. If some items (sub-items) are residue, then as per the Drug Item Reduction Plan, these items need not be analyzed.

All remaining items will be left intact should further analysis be required.

Example 1: Item 1 is submitted with 4 sub-items (1.1-1.4). All 4 items appear identical and are suspected to be methamphetamine. Combined, the total weight will be far less than 14 grams, the weight threshold of methamphetamine. In this instance, the analyst would choose the largest sub-item (item 1.2) and confirm the presence of methamphetamine. The report would indicate that item 1 consisted of 4 sub-items (1.1-1.4) that item 1.2 was selected for analysis, weighed x.x grams, and contained methamphetamine. Items 1.1, 1.3 and 1.4 were not examined. Alternatively, the weights of sub-items 1.1, 1.3 and 1.4 could also be reported, but report must be clear that only item 1.2 was confirmed.

Example 2: Item 1 is submitted with 4 sub-items (1.1-1.4). All 4 items appear identical and are suspected to be methamphetamine. Combined, the total weight will exceed 14 grams, the weight threshold of methamphetamine. In this instance, the analyst would choose as many sub-items as necessary to push the weight over the 14 gram threshold. Each item is weighed and confirmed individually. The report would

indicate the total weight, and that each item was examined individually and contained methamphetamine.

IF sub-items are not uniform and/or NOT suspected to be identical, then full analysis must be conducted.

Example 3: Item 1 is submitted with 3 sub-items (1.1-1.3). Item 1.1 contains a white powder, item 1.2 contains a tan powder, and item 1.3 contains a yellow powder. In this example, all items would be individually weighed and confirmed. If any item is residue, and analysis of the other items has yielded a measurable weight, then the residue need not be examined unless there is high likelihood that the residue is of a higher penalty group. In this example, analyst discretion and would impact the sampling plan regarding examining or not examining the residue.

Hypergeometric / Statistical Sampling: If conclusions wish to be drawn regarding the entire population of a submitted item, then the item must be sampled and examined in accordance with one of the hypergeometric statistical sampling plans listed below. HETL will utilize 2 hypergeometric sampling plans. Commonly referred to as 95/90 and 95/50.

Sampling of Multiple Units:

A. First determine the populations present in an item.
    1. Evaluate the number of units present in an item carefully.
    2. Visually inspect each of the units in the item carefully as well as any contents for homogeneity in size, color, packaging, markings, labeling and other characteristics. For analysis purposes, each intact piece of blotter paper shall be considered a unit.
    3. If after careful visual inspection it is determined that the contents of the units are homogenous, the population shall consist of all the units.
    4. If there are differences, segregate the units into individual groups, based upon such observed differences. Each group shall be analyzed as a separate population.
    5. If in the course of analysis, it becomes apparent that the population is not homogenous, new populations may be formed based upon individual chemical test results.

B. Determine the net weight for the population (If needed).
    1. For smaller populations with packaging, this can be accomplished by obtaining a net weight of each unit and summing for a total net weight.
    2. For larger populations, an estimate for net weight can be calculated using a gross weight of all units (including packaging) and a total tare weight based on the tare weight of one or more packages).

C. Determine whether the net weight exceeds a statutory threshold. If so, the administrative sampling plan may be more appropriate and cost effective for the customer.

D. If the net weight does not exceed a threshold, or if the case is being charged as a trafficking offense based on the number of dosage units (and not a weight threshold), and/or the customer wishes to conclude that more than the number of packages actually tested contains a controlled substance, then an appropriate number of items will be randomly selected from each population and fully analyzed to confirm the presence (or absence) of any controlled substance. See tables A and B below for specific numbers of samples to be examined based on the total number of items within each population.

E. The difference between the 2 statistical sampling plans (Table A and B) relates to statistical significance that can be associated with each. An example of terminology that appears on reports using each is included in an example immediately following each table:

Table A: (95 /90) This sampling plan assures with 95% confidence that at least 90% of the entire population contains the substance identified in the sample.

Number of Units	Number sampled
10	8
11-20	12
21-30	15
31-40	18
41-50	19
51-60	20
61-70	21
71-80	22
81-100	23
101-200	26
201-400	27
401-1,000	28
1,001-10,000	29

Reports issued using the 95/90 hypergeometric sampling plan shall include some reference to the total number of items submitted in each population, the number fully tested, and a statement related to the statistical significance that can be attached to the analysis.

For example: Item 1 consists of 55 small, clear plastic bags containing a tan powder, and the customer wishes to determine if all bags contain a controlled substance. According to TABLE A: (95/90 sampling plan), if all bags are homogenous, then 20 bags would be chosen at random and fully confirmed.

The report would include terminology indicating that 55 bags were submitted, 20 bags were chosen at random and found to contain 'xxx'. Statistical analysis supports that with a 95% level of confidence, at least 90% of the population contains the substance identified.

Table B: (95/50) This sampling plan assures with 95% confidence that at least 50% of the entire population contains the substance identified in the sample

Number of Units	Number sampled
5-12	3
13-66	4
>67	5

Reports issued using the 95/50 hypergeometric sampling plan shall include some reference to the total number of items submitted in each population, the number fully tested, and a statement related to the statistical significance that can be attached to the analysis.

For example: Item 1 consists of 55 small, clear plastic bags containing a tan powder, and the customer wishes to determine if all bags contain a controlled substance. According to TABLE B: (95/50 sampling plan), if all bags are homogenous, then 4 bags would be chosen at random and fully confirmed.

The report would include terminology indicating that 55 bags were submitted, 4 bags were chosen at random and found to contain 'xxx'. Statistical analysis supports that with a 95% level of confidence, at least 50% of the population contains the substance identified.

Table C refers to the required sample size to guarantee with 95% confidence that at least 50% or 95% of the population contains controlled substance if it is expected or determined that 1 or 2 sampled units do not contain controlled substances.

Table C: number of samples required to be examined if 1 or 2 units do not contain controlled substance:

Population Size N	50% of Population		90% of Population	
	1 Neg	2 Neg	1 Neg	2 Neg
10	5	5	10	-
11-20	6	8	17	20
21-30	7	9	22	27
31-40	7	9	26	32
41-50	7	10	29	36
51-60	7	10	31	39
61-70	7	10	32	41
71-80	7	10	34	43

81-90	7	10	35	45
91-100	7	10	36	46
101-200	8	10	40	53
201-300	8	10	42	55
301-400	8	11	43	57
401-600	8	11	44	58
601-800	8	11	44	59
801-1000	8	11	45	59
1001-5000	8	11	46	61
5001-10000	8	11	46	61

References specific to Sampling

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C. IDENTIFICATION REQUIREMENTS

Minimal Requirements for Complete Identification

Category A	**Category B**	**Category C**	**Cannabis Only**	**Category D**
Mass Spectrometry	Thin Layer Chromatography	Color Tests	Macroscopic Examination	PDR
Infrared Spectrometry	Gas Chromatography	Ultraviolet Spectroscopy	Microscopic Examination	Med. Scan
Raman Spectroscopy	High Performance Liquid Chromatography	Fluorescence Spectroscopy		Poison Control
				Other Reference Material (e.g. Drug Identification Bible, Logo Index, Drugs.com)

1. When a Category A method is used, then <u>at least</u> one other technique (from either Category A, B, C or D) must be used. In the case of a hyphenated method (i.e GC/MS) a third method from Category A, B, C or D must be employed. A technique is considered Category A when the data provides structural information, has a high level of selectivity and is reviewable.

   A Category A technique may not be sufficient when:

   - The method limits the ability to differentiate the analyte from a structurally similar compound or related compounds
   - The state of the sample limits the ability to distinguish the analyte of interest (i.e. mixtures or physical condition of the sample)
   - The quantity or concentration of the sample is insufficient, and all acceptability criteria are not met

2. When a Category A method is not used, then <u>at least</u> three different methods based on different analytical principles must be employed. Two of the three methods must be from Category B.

3. Cannabis exhibits tend to have characteristics that are visually recognizable. Thus, a macroscopic and/or a microscopic examination of cannabis will be considered as Category B tests when observations include documented details of botanical features. Additional testing must include at least two other techniques from Category A, B, or C. Cannabis cannot be classified as marijuana using the current analytical techniques and would need testing performed by an outside laboratory for marijauana confirmation.

4. For exhibits of cannabis (e.g. extracts or residues) that lack sufficient observable macroscopic and microscopic botanical detail, D^9-tetrahydrocannabinol (THC) must be identified utilizing the techniques in Category A and B or A and C.

5. For tablet/capsules exhibits, at least one test from Category A and at least one from either Category D or B. Markings on a tablet or capsule will be written in the case notes along with a general description of the tablet/capsule (color, size, etc.). The number or weight of the sample will be written in the case notes.

   If the sample cannot be identified by the literature treat the sample the same as an unknown sample and use the customary analytical procedures to identify the drug.

   If the sample appears to be altered, counterfeit or homemade, check the contents of the capsule or tablet as above.

   NOTE: References for analyses of drugs come from various sources, including the DEA Laboratory Manual, Clarkes' Isolation and Identification of Drugs, and various periodicals

For the use of any method to be considered of value, the test must be considered "positive." While "negative" tests provide useful information for ruling out the presence of a particular drug/drug class, these results have no value toward establishing the forensic identification of a drug substance.

At a minimum, at least one of the methods utilized within the analytical scheme must provide data that is reviewable. Some examples of reviewable data include printed chromatograms, photographs, photocopies of results, or detailed descriptions of morphological characteristics (for cannabis only).

When sample size allows, a minimum of two samplings should be used. A different analytical technique should be applied on the separate sampling for quality assurance purposes. If the sample size is limited, additional measure should be taken to assure that the results correspond to the correct sample.

If a conclusion cannot be drawn based upon the acquired results the sample will be reported as inconclusive and the reason why a conclusion could not be reached must be listed on the Certificate of Analysis.

REPORTING RESULTS:

Results will be phrased with terminology that is clear and precise to the reader of the report (i.e., Customer) Terminology such as:

"_____ was identified…"

Or

"_____ contain(s)…"

Or

"Quantity insufficient"

Reported results will include the manner of testing, i.e.:

"Method(s) of analysis: _____"

2. Minimal Requirements for Preliminary Identification

a. General Powders and Residues: At least one positive test from category B or C

b. Tablets/Capsules: At least one identification from Category D, if possible.

Category B	**Category C**	**Category D**
Thin Layer Chromatography	Color Tests	PDR
Gas Chromatography	Ultraviolet Spectroscopy	Med. Scan
Microcrystalline Tests	Fluorescence Spectroscopy	Poison Control
		Other Reference Material (e.g.: Drug Identification Bible, Logo Index, Drugs. com)

REPORTING RESULTS:

Report Tablets/capsules as:

"Description is consistent with _____"

Reported results will include the manner of testing, i.e.:

"Method(s) of analysis: _____"

IV. CASE DOCUMENTATION

A. CASE NOTES

The minimum information, which must be contained in the case notes are:

- Laboratory Identification Number
- Start Date
- General Description of Evidence and Packaging
- Condition of packaging (sealed with initials, sealed without initials, or unsealed)
- Initial Sealed Package Weight (prior to opening container)
- Analyst's signature/Initials
- Balance used
- Gross Weight (when applicable)
- Net Weight or count (when applicable)
- Sampling date and time (when applicable)
- Reserve Weight or count of material (when applicable)
- Number of weighing events, when applicable
- Evidence Sampling Plan (Note if less than two aliquots taken from the sample)
- Solvent and Lot used
- UoM
- Post Sealed Package Weight (after processing)
- Qualitative Analysis Results
- Summary of Findings
- Date Sealed
- End Date

All case notes, spectra and other data generated during analysis will bear the initials of the analysts and the case number.

B. CASE FILE

The minimal information, which must be contained in the individual case file consists of:

- Copy of the final report/Certificate of Analysis
- Case Review Form (technical/administrative)
- Any preliminary, supplementary, or corrected reports
- Solid Dose Drug Worksheet and Case notes
- Evidence Receipt/Contract for Examination form
- Hard copies of data that support the conclusion of the analyst.
- Other: Discrepancy form / correspondence with customer, (if applicable)
- Chain of Custody form

V. GENERAL ANALYTICAL PROCEDURES

NOTE: There are times when deviations from documented policy or procedure is/are necessary. These must be communicated to the customer on the Certificate of Analysis. Refer to the Section's Quality Manual for description and protocols to request deviations.

A. GAS CHROMATOGRAPHY/MASS SPECTROMETRY

1. SAMPLE PREPARATION

- Add approximately 1 ml of SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, to a 2 ml GC/MS vial.
- Add sufficient amount of sample (varying on suspected concentration of drug being tested) from the test tube to the same GC/MS vial and cap. Add the same volume of the procedure blank to the corresponding GC/MS vial. See VI. Specific Analytical Procedures section for sample solvent guidance.
- Repeat for each sample and procedure blank, if applicable.
- Prepare an instrument blank (1ml SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, and an equivalent volume of the same solvent used in the extraction) to be run between each sample. Ensure the solvent concentration is greater than or equal to the concentration used in the sample to screen for contamination.
- Run GC/MS analysis using the appropriate method (Rapidrug, WBAMINE2, STEROID, etc…).

2. Criteria for Evaluating Unknowns

Unknown samples will be evaluated for suitability prior to comparison to known standard. To determine if the unknown sample is suitable for comparison the analyst will evaluate and consider each of the following:

A) Absence of target compound(s) as listed on the Quant QEdit Report for the method used and/or other compounds at the discretion of the analyst in the blank prior to the sample
B) Presence of the internal standard peak with a signal to noise ratio > 3:1

If the sample is deemed suitable for comparison the analyst can determine the presence of a controlled substance and/or diluent. To determine the presence of a controlled substance and/or diluent on the GC/MS, the analyst will evaluate and consider each of the following:

A) Ratios of Target Ion and Qualifying Ions *between the known (standard) and unknown* should be evaluated.
B) Comparison of the retention times. The sample peak shall be within +/- 0.25 mins. of the reference peak.
C) Comparison of the unknown spectra to the reference spectra, using any approved library, with a minimum quality match of 80 and/or the discretion of the analyst.
D) Signal to noise ratio >3:1

C. FOURIER TRANSFORM INFRARED SPECTROSCOPY

The following outlines the sample prep and analysis for the Thermo Fisher Nicolet iS20 ATR FTIR.

Launch OMNIC by double-clicking the icon on the desktop. Verify that the "Solid Dose Drug" experiment is selected (visible at the top of the window).

Analyzing Sample Blank

- Move the sample arm to the upright position and turn it to the side. Note: to lower the arm, turn the knob clockwise. To raise the arm, counterclockwise. Should the arm become locked in the up position, meaning you turn the knob and it will not lower, manually move the arm slightly and turn the knob clockwise until the mechanism engages.
- Clean the tray and crystal area by adding solvent to a kimwipe and wiping down the area. AT NO TIME SHOULD CANNED AIR BE USED ON THIS INSTRUMENT IN ANY FORM.
- Click "Col Smp" Button > it will prompt you to enter spectrum title (case and sample number followed by "blank")
- Click "ok" > it will prompt you to run background
- Click "ok" > to initiate background > click start collection button (upper right corner)
- After background is complete, Click "ok" > lower the arm down until it gently touches the crystal (without sample and w/o locking the arm)
- Click "start collection" > once complete the confirmation box appears > click "YES"
- Save the file by clicking File > Save as > then enter your filename or click the "Set Filename to Title" button. Files shall be saved in the appropriate year folder found in My Documents > OMNIC > Spectra > SDD Analysis
- Evaluate the spectra to ensure it is absent of distinct peaks. If suspected peaks are present, click "Find Pks" button to label peaks. Should some peaks not label, adjust the sensitivity bar on the left of the screen to increase the level and further display peaks.
- Once you are satisfied with the labeling, click the "Replace" button to update your active window. Click "full scale" button to automatically adjust peak heights and labels to fit in the window. Click save.
- If there are peaks present that are similar to expected peaks in the sample to be analyzed, perform the cleaning procedure again and analyze a new blank prior to proceeding with sample analysis. Both blanks shall be maintained for the casefile.

Analyzing Samples and Performing Library Search

- Move the sample arm to the upright position and turn it to the side. Note: to lower the arm, turn the knob clockwise. To raise the arm, counterclockwise. Should the arm become

locked in the up position, meaning you turn the knob and it will not lower, manually move the arm slightly and turn the knob clockwise until the mechanism engages.
- Clean the tray and crystal area by adding solvent to a kimwipe and wiping down the area. AT NO TIME SHOULD CANNED AIR BE USED ON THIS INSTRUMENT IN ANY FORM.
- Click "Col Smp" Button > it will prompt you to enter spectrum title (case and sample number)
- Click "ok" > it will prompt you to run background
- Click "ok" > to initiate background > click start collection button
- After background is complete > Click "ok"
- Add a small amount of sample to the crystal and lower the arm down until it locks in place
- Click "start collection" > once complete the confirmation box appears > click "YES"
- Click "Find Pks" button to label peaks. Should some peaks not label, there is a sensitivity bar on the left of the screen that can be increased to further display peaks.
- Once you are satisfied with the labeling, click the "Replace" button to update your active window. Click "full scale" button to automatically adjust peak heights and labels to fit in the window.
- Save the file by clicking File > Save as > then enter your filename or click the "Set Filename to Title" button.
- With both the blank and appropriate sample still open, click the "Stack Spe" button from the toolbar.
- With the sample selected (spectra line will be red if it is the active selection) click "Search" button > the library search should open up with the appropriate hits for the libraries selected
- Click on the appropriate match > click "Overlay" button down below
- If satisfied click "window" on the top menu and select the window with the current sample and blank displayed. (Window 1/Window 2/etc…).
- Once the correct window is displayed showing the current blank and sample, click "report" in the top menu.
- Click print/preview to load the SDD Report Template and view the preview.
- Ensure the correct data is displayed and the correct library match is loaded.
- Click Print
- If an acceptable library match has not been achieved, close the search window and perform a subtraction as outlined below.

Should a zoom be required the traditional right-click and drag can be used or you can use the spectra window at the bottom of the screen to adjust what portion of the spectra is visible. To reset the zoom to the previous setting there are two easy ways of doing so. 1) double left-click in the white portion (the selected portion) of the spectra window at the very bottom or 2) select the View menu and select "Undo Limit Change"

The libraries approved for casework comparisons are:
- SDD Library
- Master SFL1 FTIR library v. 082719
- HR Georgia State Forensic Drugs

Performing Subtraction:
Should a low quality match be attained on a specific sample, an analyst can subtract a known reference spectra from an approved library to improve the unknown spectra.

- Ensure sample spectra is open
- Open the reference spectra to subtract
    - Click the "Lib Mgr" button on the toolbar. Select the desired search library (or use the search spectra function). Select the desired compound. Click the "Add to Window" button. Scroll down the list of windows to add the spectra to the target window and click the "Add" button.
- Click on your unknown spectra (should have a red border) and then also select the spectra to subtract by holding the Ctrl button while selecting the second spectra (both should have a red border)
- Go to Process > Subtract
- Click the "Add" button in the upper right corner to add the new spectra to a new window.
- Click "Search" button > the library search should open up with the appropriate hits for the libraries selected
- Click on the appropriate match > click "Overlay" button down below
- Click "window" on the top menu and select the window with the current sample and blank displayed. (Window 1/Window 2/etc…).
- Once the correct window is displayed showing the current blank and sample, click "report" in the top menu.
- Click print/preview to load the SDD Report Template and view the preview.
- Ensure the correct data is displayed and the correct library match is loaded.
- Click Print
- Save the subtraction file by clicking File > Save as > then enter your filename or click the "Set Filename to Title" button and add subtraction to the end of the filename.

Criteria for Evaluating Unknowns

Unknown samples will be evaluated for suitability, prior to comparison to known standard, by evaluation of the blank and the sample. The maximum scan range permitted is 4,000-400 wavenumbers, however this can be adjusted to narrower range to remove excessive noise peaks in the lower wavenumber region, as long as all required peaks for the compound in the tables listed below are still present in the spectra.

To determine if the unknown sample is suitable for comparison the analyst will evaluate and consider each of the following:

A) Ensure the blank was run using a resolution of 4 and run at 16 scans. Evaluate the blank run prior to evaluation of the sample to ensure the blank is clean and free of any obvious interference and/or background noise. A clean blank is defined as a blank run with the same scan range as the sample and having all peaks beyond the noise region having a response below 0.05, or if there are peaks present above 0.05 the peaks will be labeled and no peaks of interest will be present. If the blank is deemed suitable, move on to evaluation of the sample.

B) Evaluate the overall appearance of the sample spectra and ensure it is free of any obvious interference and/or background noise. Ensure the sample was run using a resolution of 4 and run at 16 scans.

If the sample is deemed suitable for comparison the analyst can determine the presence of a controlled substance and/or diluent. To determine the presence of a controlled substance and/or diluent on the FTIR the analyst will evaluate and consider each of the following:

A) Overlay and compare with the reference spectra and achieve a library match to an approved library of 80 or greater.

B) At least 5 significant peaks that help to identify the compound should agree within +/- 4 cm-1 of known reference peaks. Significant peaks for commonly encountered drugs are listed below and shall be used for comparison. All peaks must be present within the unknown sample before it is deemed 'confirmed' by FTIR.

Cocaine Base	
Peak Number	**Peak at Wavenumber cm-1**
1	712
2	1106
3	1036
4	1275
5	1450
6	1706
7	1734

Cocaine Hydrochloride	
Peak Number	**Peak at Wavenumber cm-1**
1	730
2	1105
3	1230
4	1265
5	1712
6	1728

Methamphetamine Hydrochloride	
Peak Number	**Peak at Wavenumber cm-1**
1	698
2	747
3	1059
4	1453
5	1486
6	1603

Amphetamine	
Peak Number	**Peak at Wavenumber cm-1**
1	696
2	738
3	1454
4	1496
5	1513
6	1573

Pseudoephedrine	
Peak Number	**Peak at Wavenumber cm-1**
1	543
2	630
3	700
4	1006
5	1373
6	1454

Ephedrine	
Peak Number	**Peak at Wavenumber cm-1**
1	671
2	697
3	750
4	991
5	1388
6	1453

Phentermine	
Peak Number	**Peak at Wavenumber cm-1**
1	447
2	702
3	727
4	1284
5	1389
6	1610

α-PVP	
Peak Number	**Peak at Wavenumber cm-1**
1	718
2	770
3	1232
4	1337
5	1449
6	1681

For drugs other than those listed above, confirmation criteria shall include a library match score of 80 or greater to the HETL library, created in house using reference material purchased from an approved vendor, and at least five prominent peaks within +/- 4 cm^{-1} are present when compared with a published reference spectra from SWGDRG, Cayman Chemical, Cerilliant, or other similar source.

Printed results must include the number of sample scans, the number of background scans, the resolution, the name of the sample, the date the sample was analyzed and the date the report was printed.

Adding Compounds in the SDD Library

With the spectra selected, click “Add Lib” button on the toolbar. A window will open, select the “HETL SDD” library and click “OK”. In the next window, enter the requested information for the spectra (Name, Manufacturer, Lot, Exp) and then click “OK”.

Deleting Compounds to the SDD Library

Click the “Lib Mgr” button on the toolbar. Select the “HETL SDD” library from the search libraries. Select the spectra to be deleted and click the “Delete” button. The compound name will still be visible in the index and an “X” will be displayed in the delete column.

VI. SPECIFIC ANALYTICAL PROCEDURES

A. Cannabis/Hashish

Chemicals:

SDD Solvent Mix
6N HCL

Safety Precautions and PPE:

Lab coats, gloves and eye protection will be worn when handling chemicals in accordance with the SDS.

Use respiratory protection when handling moldy samples**.**

ANALYSIS:

1. Weight/Count

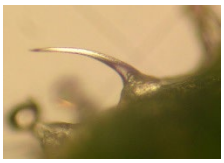
Obtain the total net weight of the plant/resinous material or total count of whole plants and note the weight/count in case notes. Report all weights as net weights in grams (or pounds) on worksheet and report/Certificate of Analysis.

2. Microscopic

The identification of cannabis depends largely on identifying its botanical features. Cannabis is characterized by the presence of cystolithic, glandular and non-glandualar hairs on the leaf.

A. Cystolithic Hairs

Cystolithic hairs are claw-shaped, usually curved structures with a broad circular base and are mostly one-celled. Within the hair is a cystolith of calcium carbonate. The presence of a true cystolithic should be confirmed by the addition of hydrochloric acid.



- Place a small amount of plant material on a glass slide.
- Add a few drops of water and cover with a coverslip.
- Add a few drops of 6 N Hydrochloric Acid to the side of the coverslip.
- Evolution of bubbles of carbon dioxide confirms the presence of a true cystolith.

B. Glandular Hairs

Glandular hairs are important because they contain and secrete resin. They are short and may be either unicellular or multicellular. The bigger glandular hairs have a multicellular stalk with heads containing 8 to 16 cells.

C. Non-glandular Hairs

On the reverse side of the leaf containing the cystolithic hairs can be found numerous long, wide nonglandular covering hairs.

A positive microscopic exam will include the observation/presence of **all three types** of hairs.

- Examine the plant material under microscope.
- Look for presence of the botanical features (hairs), indicative of Cannabis and record in the case notes.

3. Extraction for GC/MS

- To a small amount of dried plant material in a dish or test tube add approximately 2-5 mL SDD Solvent Mix or other solvent. Obtain a second empty dish or test tube to serve as an extraction procedure blank and add the same amount of solvent to the dish. The resin from the plant, which contains THC and other Cannabinoids, will be suspended in the solvent.
- Shake the tubes for the samples and extraction procedure blank and let sit for approximately one minute. Centrifuge if needed.
- If necessary, concentrate the sample and extraction procedure blank by placing the test tube under N_2 gas to evaporate a portion of solvent.

4. Gas Chromatography/Mass Spectrometry

Use a portion of the liquid in the test tube for a gas chromatography/mass spectrometry (GC/MS) confirmation analysis.

- For instrument blank: Fill GC/MS vial with approximately 1mL of appropriate solvent and add TPP or DFTPP Internal Standard, if internal standard is not already included in the solvent solution.

- For Sample and extraction procedure blank: Fill GC/MS vial with approximately 1mL of appropriate solvent and add TPP or DFTPP Internal Standard, if internal standard is not already included in the solvent solution. Add the same volume of sample and extraction procedure blank to the GCMS vials.

- Run all samples on THC, Rapid Drug or Steroid method on GC/MS to determine the presence of Delta-9 THC. When possible, the use of two different methods, solvents, or instruments, should be used for confirmation purposes.

5. Reporting

The laboratory currently does not perform quantitation of delta-9 THC and CBD to differentiate marijuana from hemp. As such, if the sample includes a positive microscopic identification and confirmation of delta-9 THC the report shall state: "Plant-like material identified as cannabis and contains delta-9 THC. Further analysis would be required to differentiate the material as marijuana or hemp."

B. General Powders/Residues

Chemicals:

Hexanes	Acetonitrile	9.5 pH Ammonium Buffer		Isopropanol
Chloroform	Ethyl Acetate	Methanol	Acetone	Methylene Chloride
DFTPP	95% Ethanol	NaOH	NH4OH	HCl

Safety Precautions and PPE:

Lab coats, gloves and eye protection will be worn when handling chemicals in accordance with the SDS.

When handling hypodermic apparatuses, the analyst will remove caps employing mechanical means such as tweezers. Once removed, the caps will not be replaced back on the hypodermic apparatus. Instead, place cap and needle back into the submitted biohazard container.

ANALYSIS:

1. Weight

Obtain the total net weight of the material and note the weight in case notes. Report all weights as net weights on worksheet and Report/Certificate of Analysis.

2. Sample Prep and Extraction

Powders:

When applicable, two aliquots will be taken for separate tests. Powders will be dissolved in SDD Mix A/B/C, methanol, or another appropriate solvent depending on suspected substance (consult the Merck Index, Clarke's Isolation and Identification of Drugs, or other appropriate reference for solubility and chemical property information). A portion of the dissolved sample can then be used for GCMS analysis. A separate portion of the powder can be used for FTIR analysis.

For powders which contain mixtures, either liquid-liquid extraction using water and an appropriate immiscible solvent, or an acidic/basic extraction can be used (consult the above listed references).

To determine the most appropriate solvents:

- The greater the log P value of the target compound, the more likely the solute will extract into an organic solvent
- Distribution of neutral compounds can be estimated based on their solubility in different solvents
- Use the reference pK_a value of the drugs to determine the solvents and pH needed for the extraction. Only undissociated compounds will extract into the organic phase. Compounds are ~50% ionized at a pH value equal to the pK_a.
    - Acidic compounds reach ~100% ionization at >2 pH units above the compound's pK_a
        - ~1% ionization is observed at >2 pH units below the compound's pK_a
    - Basic compounds reach ~100% ionization at >2 pH units below the compound's pK_a
        - ~1% ionization is observed at >2 pH units above the compound's pK_a
    - Weakly acidic or weakly basic
        - Maximum yield will be seen 2 pH units below (for acidic compounds) or 2pH units above (basic compounds)
- pK_a of basic drugs can be determined by the following equation
    - $pK_a = pK_w - pK_b$
        - pK_w is the negative decimal logarithm of the dissociation constant of water and equals 14 at 25^oC
- When separating acidic and basic drugs, sulfuric acid is preferred over hydrochloric acid as many hydrochloride drugs are soluble in organic solvent
- Ammonium solution will only adjust an aqueous solution to a pH of ~10, which may not be sufficient for some drugs

- Drugs that have one acidic and one basic function (amphoteric) have yields that are more difficult to predict. They can be extracted as non-ionized compounds with a pH that allows for the ionization of the basic group to have ended but the ionization of the acidic group has not yet started.
    - The pK_a and pK_b should be added to for a total to determine the point where the molecule would not be ionized. The total is then subtracted from the pK_b to determine the basic function's pK_a. A pH value 1-2 units above the basic function's pK_a and 1-2 units below the acidic function's pK_a should be used for extraction. Yields will vary depending on the strength of the acid and base functions and the ability of the molecule to ionize at the selected pH.

When multi-solvent (complex) extractions are performed an extraction procedure blank will be utilized. An extraction procedure blank sample shall be run as an extraction blank alongside samples following the same procedure listed below. All extraction procedure blanks, instrument blanks, and sample data will be included in the casefile.

Acid/Neutral Extraction Procedure:

1. Add DI water to portion of the sample
2. Acidify the sample using an appropriate acid, to the appropriate pH
3. Add a similar volume of an appropriate organic solvent
4. Vortex the mixture and spin down
5. Pull off the organic phase into a new tube. The remaining aqueous phase will contain any basic and water soluble drugs and should be reserved for basic extraction, if basic drugs are suspected.
    a. This organic phase should contain most acidic and neutral drugs
    b. To further separate neutral drugs from acidic drugs:
        i. Add a similar volume of 0.1N NaOH to the organic phase
        ii. Vortex the mixture and spin down
        iii. Remove organic phase into a new tube
            1. This phase should have neutral drugs
            2. Dry down for analysis
    c. The remaining aqueous phase will contain sodium salts of acidic drugs. To extract the acidic drugs:
        i. Adjust the pH of the aqueous phase to 1-3
        ii. Add a similar volume of organic solvent
        iii. Vortex the mixture and spin down
        iv. Remove organic layer into a new tube, dispose of aqueous phase
            1. This tube should have acidic drugs
            2. Dry down for analysis

Basic Extraction Procedure:

1. Utilize aqueous phase from step 5 above if continuing from an acid/neutral extraction. If only performing a base extraction, add DI water to portion of the sample
2. Adjust the pH of the sample using an appropriate base, to the appropriate pH needed for the suspected drug
3. Add a similar volume of an appropriate organic solvent
4. Vortex the mixture and spin down
5. Pull off the organic phase into a new tube. The remaining aqueous phase should be discarded.
    a. This organic phase should contain most basic drugs
    b. Dry down for analysis

Residues:
Residual samples where no weighable amount of sample is present, or the sample is present at a weight below the most current estimated uncertainty of measurement, should be noted in the case notes. The sample/item which contains the residue can either be rinsed or swabbed with SDD Mix A/B/C, methanol, or an appropriate solvent and that sample used for GC-MS analysis. If the residue contains a mixture the sample can be rinsed/swabbed with water and either a liquid-liquid, acidic, or basic extraction used.

3. Gas Chromatography/Mass Spectrometry – (refer to Section V. GENERAL ANALYTICAL PROCEDURES – Part B)

Use a portion of the remaining extracted sample in the test tube for a gas Chromatography/ Mass Spectrometry (GC/MS) confirmation analysis.

- Add approximately 1 ml of SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, to a 2 ml GC/MS vial.
- Add sufficient amount of sample (varying on suspected concentration of drug being tested) from the test tube to the same GC/MS vial and cap. Add the same volume of the procedure blank to the corresponding GC/MS vial.
- Repeat for each sample and procedure blank, if applicable.
- Prepare an instrument blank (1ml SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, and an equivalent volume of the same solvent used in the extraction) to be run between each sample. Ensure the solvent concentration is greater than or equal to the concertation used in the sample to screen for contamination.
- Run GC/MS analysis using the appropriate method (Rapidrug, WBAMINE2, STEROID). When possible, the use of two different methods, solvents, or instruments, should be used for confirmation purposes.

4. FTIR

Use a separate aliquot, if possible, for FTIR confirmation analysis.

Refer to Section V. GENERAL ANALYTICAL PROCEDURES – Part C for analysis by Fourier Transform Infrared Spectroscopy.

C. TABLETS/CAPSULES/FILMS

Chemicals:

Hexanes	Isopropanol	Methanol	NaOH
DFTPP	Methylene Chloride	95% Ethanol	Acetone
Acetonitrile	Ethyl Acetate	NH_4OH	Chloroform

Safety Precautions and PPE:

Lab coats, gloves, and eye protection will be worn when handling chemicals in accordance with the SDS.

ANALYSIS:

1. Weight/Count

Obtain the total count of the tablets/capsules/Films and record in case notes. Obtain the net weight of at least one item and record it in the case notes.

2. Extraction

Extraction A **Low Concentration Prescriptions** **Lisdexamfetamine**	**Extraction B**
1. Crush tablets(s), empty capsule, or sample strip and place portion of sample in a test tube. Repeat for each sample being tested. 2. Prepare extraction procedure blank 3. Perform an acidic/basic/neutral extraction on extraction procedure blank and all samples in extraction set 4. A portion of the extracted samples and extraction procedure blank can then be used for GCMS analysis. **IF NECESSARY:** 5. Dry down sample and procedure blank	1. Crush tablet(s) and place in a test tube. 2. A portion of the sample can then be used for GCMS analysis.

3. Gas Chromatography/Mass Spectrometry – (refer to Section V. GENERAL ANALYTICAL PROCEDURES – Part B)

Use a portion of the remaining sample in the test tube for a gas chromatography/mass spectrometry (GC/MS) confirmation analysis.

- Add approximately 1 ml of SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, to a 2 ml GC/MS vial.
- Add sufficient amount of sample (varying on suspected concentration of drug being tested) from the test tube to the same GC/MS vial and cap. Add the same volume of the procedure blank to the corresponding GC/MS vial.
- Repeat for each sample and procedure blank, if applicable.
- Prepare an instrument blank (1ml SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, and an equivalent volume of the same solvent used in the extraction) to be run between each sample. Ensure the solvent concentration is greater than or equal to the concertation used in the sample to screen for contamination.
- Run GC/MS analysis using the appropriate method (Rapidrug, WBAMINE2, STEROID). When possible, the use of two different methods, solvents, or instruments, should be used for confirmation purposes.

4. FTIR (If necessary) – (refer to Section V. GENERAL ANALYTICAL PROCEDURES – Part C)

Use a portion of the remaining sample in the test tube for FTIR confirmation analysis**.**

- Dry the sample on a watch glass
- Run FTIR analysis (Refer to Section IV. GENERAL ANALYTICAL PROCEDURES – Part D for analysis by Fourier Transform Infrared Spectroscopy)

D. SUSPECTED LSD

Samples suspected of containing lysergic acid diethylamide (or lysergic acid methylpropylamide) need to be handled carefully when being analyzed. LSD will most often be placed on paper, small gelatin squares, and very small homemade tablets or in a liquid.

Chemicals:

Concentrated HCL Methanol Chloroform DFTPP Ethanol

Safety Precautions and PPE:

Lab coats, gloves, and eye protection will be worn when handling chemicals in accordance with SDS.

ANALYSIS:

1. Weight/Count

Obtain the total number of dosage units (perforated squares on paper, number of gelatin squares, etc.). The number of dosage units will be reported as such on the final report/Certificate of Analysis.

2. Extraction

Note an extraction procedure blank sample shall be run as an extraction blank alongside samples following the same procedure listed below. All extraction procedure blanks, and sample data will be included in the casefile.

- After determining the number of dosage units, prepare for the analysis of the sample by placing an appropriate number of dosage units in a disposable glass test tube.
- Prepare an empty tube to be used as an extraction procedure blank
- Add to both the sample and extraction procedure blank approximately 100-500ul of methanol and let stand for approximately fifteen minutes.
- While the LSD is being extracted into the methanol, check the liquid in the test tube for fluorescence in the viewing box while using the long wave UV lamp. Fluorescence is indicative of LSD and/or LAMPA.
- A portion of the dissolved sample can then be used for GCMS analysis.

3. Gas Chromatography/Mass Spectrometry – (refer to Section V. GENERAL ANALYTICAL PROCEDURES – Part B)

Use a portion of the remaining sample in the test tube for a gas chromatography/mass spectrometry (GC/MS) confirmation analysis.

- Add approximately 1 ml of SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, to a 2 ml GC/MS vial.
- Add sufficient amount of sample (varying on suspected concentration of drug being tested) from the test tube to the same GC/MS vial and cap. Add the same volume of the procedure blank to the corresponding GC/MS vial.
- Repeat for each sample and procedure blank, if applicable.
- Prepare an instrument blank (1ml SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, and an equivalent volume of the same solvent used in the extraction) to be run between each sample. Ensure the solvent concentration is

greater than or equal to the concertation used in the sample to screen for contamination.

- Prepare LSD or LAMPA standard by adding 10µl of a CRM to 100-300 µl SDD Mix C in an insert.
- Run both standard and sample by GC/MS analysis using the LSD method and the Steroid method for confirmation. When possible, the use of two different methods or instruments, should be used for confirmation purposes.

E. PSILOCYBE MUSHROOM

Psilocybe mushrooms are relatively small mushrooms that contain the hallucinogenic compounds psilocin and psilocybin. This is a complex biological matrix, requiring extraction prior to analysis.

NOTE: If the mushroom samples are fresh, they must be dried prior to analysis..

Chemicals:

6% Glacial Acetic Acid Dichloromethane Ammonium Hydroxide Chloroform

Safety Precautions and PPE:
Lab coats and eye protection will be worn when handling chemicals in accordance with the SDS.

ANALYSIS:
1. Weight

Obtain the total net weight of the material and note the weight in case notes.

2. Extraction Option 1

Note an extraction procedure blank sample shall be run as an extraction blank alongside samples following the same procedure listed below. All extraction procedure blanks, and sample data will be included in the casefile and documented on the mushroom extraction worksheet that can be found on SharePoint.

- Prepare an extraction worksheet listing all blanks and samples that will be extracted
- Prepare a 6% by volume glacial acetic acid solution
- Weigh out approx. 2.0 grams of mushrooms
- Break-up the dried mushrooms into smaller pieces
- Place the mushroom pieces into a dish or beaker. Prepare an empty dish or beaker to serve as an extraction procedure blank. Add the 6% acetic acid solution, enough acetic acid to completely cover the small pieces, to both the sample and the extraction procedure blank.
- Let stand for at least 30 minutes, stirring occasionally

- Filter out the mushroom pieces and retain the liquid portion. Retain the liquid for the extraction procedure blank.
- Transfer the liquid portion for the sample and extraction procedure blank into separate separatory funnels for washing.
- Wash the contents of the separatory funnels with dichloromethane. Vigorously invert the funnel to mix the solvent and aqueous layer. Allow sample to sit for a few minutes and settle into two layers. Drain off the lower organic layer.
- Repeat the washing of the aqueous layer two more times with dichloromethane
- Check the pH of the aqueous layer – should be between pH 1 and 2
- Basify the solution with ammonium hydroxide until you reach pH between 9 and 10
- Wash the solution with chloroform, vigorously inverting the separatory funnel to mix the two layers. Be sure to vent the separatory funnel. Allow the sample and extraction procedure blank to sit for approximately 10 minutes. Drain and save the chloroform layer (psilocin is in the chloroform layer) into an evaporating dish. If necessary, filter the chloroform layer.
- Repeat chloroform wash two more times –combining the chloroform layers into the same evaporating dish, per sample/extraction procedure blank.
- If necessary, concentrate the sample and extraction procedure blank by placing under N_2 gas.

3. Extraction Option 2

Sampling
Fill two test tubes with approximately half an inch (thumb width) of broken mushroom material, ensuring a good representative sample. These samples will be used for the rapid screen method. Additionally, add approximately two grams of material to an appropriate vessel to be used for extraction option 1, should it be required for analysis. Obtain an empty tube to serve as a extraction procedure blank.

Rapid Screen Method:
Individual sample prep: Add SDD mix A/B/C to sample and extraction procedure blank test tubes until the solvent is just covering the material. Vortex the test tube appropriately and allow to soak. It is recommended to allow the material to soak for at least one hour. Longer soak times are more likely to present higher concentration of any analytes present in the material.

4. GC/MS Confirmation - (refer to Section V. GENERAL ANALYTICAL PROCEDURES – Part B)

Extraction Option 1:

Use a portion of the remaining sample in the evaporation dish for a gas chromatography/mass spectrometry (GC/MS) confirmation analysis.

- Add 100-200ul of MeCL2/EtOH/DFTPP solution to the residue in the evaporation dish for the samples and extraction procedure blank.

- Transfer extracts for the sample and extraction procedure blank to a GC/MS vial with an insert and cap. Add the same volume of sample extract and procedure blank extract to the corresponding GC/MS vial.
- Repeat for each sample and procedure blank, if applicable.
- Prepare an instrument blank (1ml SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, and an equivalent volume of the same solvent used in the extraction) to be run between each sample. Ensure the solvent concentration is greater than or equal to the concertation used in the sample to screen for contamination.
- Run GC/MS analysis using the appropriate method (Rapidrug).

Extraction Option 2:

- Prepare a GC/MS vials for the sample and process extraction procedure blank for analysis by adding the same volume of the sample and extraction procedure blank to each vial and then add the appropriate solvent and DFTPP internal standard, if necessary.
    - recommended preparation: in an insert, place 20 to 50 microliters of sample solvent and 100 to 150 microliters of SDD mix C.
- Prepare an instrument blank (1ml SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, and an equivalent volume of the same solvent used in the extraction) to be run between each sample. Ensure the solvent concentration is greater than or equal to the concertation used in the sample to screen for contamination.
- The blank should be run, followed by the sample, using an approved method on the GC/MS (RAPIDRUG/STER). When possible, the use of two different methods, solvents, or instruments, should be used for confirmation purposes. If a psilocin is not confirmed via the rapid screen method, extraction method 1 should be performed.

5. Microscopic Examination

For food products suspected of containing psilocybe mushrooms and cannabis/delta-9 THC microscopic examination may be necessary to assist in the analysis.

F. LIQUID SAMPLES AND FOOD PRODUCTS

Chemicals:

Chloroform	Methylene Chloride	Isopropanol
Sodium Hydroxide	Ammonium Hydroxide	Concentrated HCL
6N HCL		

Safety Precautions and PPE:

Lab coats, gloves, and eye protection will be worn when handling chemicals in accordance with the SDS.

ANALYSIS:

1. Volume

Estimate the total volume of the liquid, if possible, and note in case notes. Obtain a weight for all food products.

2. Extraction

Aqueous liquid samples can be extracted with an acidic, basic and/or neutral system using an appropriate immiscible solvent depending on the suspected or purported target analyte(s). When multisolvent (complex) extractions are performed an extraction procedure blank will be utilized.

An extraction procedure blank sample shall be run as an extraction blank alongside samples following the same procedure listed below. All extraction procedure blanks, instrument blanks, and sample data will be included in the casefile.

Solid food samples can be soaked in water and the water portion then treated as a liquid sample above. Suspected Δ9 THC food products can be soaked in SDD Mix solution. Consult the Merck Index, Clarke's Isolation and Identification of Drugs, or other appropriate reference for solubility and chemical property information.

A portion of the dissolved sample can then be used for GCMS analysis.

3. Gas Chromatography/Mass Spectrometry – (refer to Section V. GENERAL ANALYTICAL PROCEDURES – Part B)

Use a portion of the remaining sample in the test tube for a gas chromatography/mass spectrometry (GC/MS) confirmation analysis.

- Add approximately 1 ml of SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, to a 2 ml GC/MS vial.
- Add sufficient amount of sample (varying on suspected concentration of drug being tested) from the test tube to the same GC/MS vial and cap. Add the same volume of the procedure blank to the corresponding GC/MS vial.
- Repeat for each sample and procedure blank, if applicable.
- Prepare an instrument blank (1ml SDD Mix C, or other appropriate solvent with the addition of DFTPP internal standard, and an equivalent volume of the same solvent used in the extraction) to be run between each sample. Ensure the

solvent concentration is greater than or equal to the concertation used in the sample to screen for contamination.

- Run GC/MS analysis using the appropriate method (LSD, Rapidrug, WBAMINE2, STEROID). When possible, the use of two different methods, solvents, or instruments, should be used for confirmation purposes.

G. WASTE

1. Waste Stream Evaluations

- All procedures and processes involving hazardous chemical, biological or drug waste must have a waste stream evaluation form completed and on file with the Quality Manager prior to performing the procedure.
- All new chemicals/procedures must be evaluated prior to purchase to ensure an appropriate waste stream is available for disposal. See the Chemical Hygiene Plan for more of the New Chemical Procurement procedure.

2. Hazardous Solvent Waste

- All extraction waste for evaluated procedures shall be collected in the flammable liquid dual waste satellite waste container. See the Chemical Hygiene Plan and RCRA Plan for specifics relating to satellite waste containers.
- Biological waste shall be collected in the Forensic liquid dual waste satellite waste container.

3. GC/MS Vial Waste

- All GC/MS vials used to analyze seized drug evidence using evaluated procedures shall be collected in the GC/MS Vial satellite waste container. See the Chemical Hygiene Plan and RCRA Plan for specifics relating to satellite waste containers.

4. Residual Evidence Waste

- All residual waste from processing of seized drug evidence will be collected in the Rx Destroyer, or similar product, collection container. Residual evidence waste collected in the container are rendered inactive and irretrievable and may be disposed of in the regular trash or removed by an approved waste hauler.

5. Consumables used to process seized drug evidence

- Any lab consumables used to process seized drug evidence shall be disposed of in a glass waste box that is marked for MDEA disposal.
- Full MDEA glass boxes should be sealed with evidence tape and stored in the evidence suite for pick up and incineration by MDEA.

6. Reference Materials

- Any DEA non-exempt reference material must be disposed of following the procedure outline in the SOP Manual.

- Expired DEA exempt reference materials that are not empty shall be disposed of in the Forensic GC/MS vial waste satellite waste container.
- Empty DEA exempt reference material containers shall be disposed of in a glass waste box. Please reference the definition of empty found in the Chemical Hygiene and RCRA plan.

APPENDIX A

OPERATION OF THE GC-MS

GAS CHROMATOGRAPHY - MASS SPECTROMETRY

1. INSTRUMENT PREPARATION

The following is not meant to replace the manuals supplied by the instrument vendor, but rather to provide a general 'step by step' overview to the operation of the GCMS. In no way is this meant to be a detailed procedure that must be fully followed and documented in the exact order. Rather it is designed to be a resource, or 'quick reference guide' to any new employee learning MS and specifically the system currently in use at HETL.

Fill solvent wash bottles on the auto-sampler tower. There are two bottles: one for methylene chloride and one for methanol.

Tune the mass spectrometer. On the instrument control panel, click the "Tune MS icon" (tuning fork and music notes picture). Evaluate the generated tune and determine if the tune is satisfactory.

Make sure printer has paper in the paper tray.

Check the pressure of the Helium carrier gas tank. (Replace the Helium tank when the pressure is down to 500 psi. **DO NOT** drain the tank dry).

2. PREPARE THE SEQUENCE

Arrange vials in the auto-sampler tray(s).

Begin by clicking the "Write Sequence Icon" (Pencil and 3 auto-sampler vials picture) on the Instrument Control Panel.

Click the "Data Path" box at the top of the screen.

The data path designation is the following:

D:\HPCHEM\2\DATA\MMDDYY

MMDDYY is the six-digit date designation representing the day the sequence was started.

In the case that more than one sequence is run on the same date add the suffix "A".
(Example: MMDDYYA)
Continue onto the next letter of the alphabet for each subsequent sequence begun on that day.

To add a date to the list of data folders, highlight the "2" in the menu and click the box "Make New Folder".

Fill in the Sample Log Table using the following table as a template.

Type	Vial	Data File	Method	Sample	Multiplier	Comment
SAMPLE	Begin with first vial no.	Begin with 001	RAPIDRUG STER WBAMINE2 Etc.	BLANK, CASE/ SAMPLE NO., STANDARD	1.0000 or Calculated value for quantitation	Lot No.

Complete a row for each vial on the auto-sampler. Use the highlight and right click features for cutting, pasting, copying, and repeating row function.

When the Sample Log Table is complete, CLICK OK.

Click the "Save Sequence Icon" (3 ¼ inch Floppy Disk and 3 auto-sampler vials picture).
Save the sequence with the six-digit date designation matching the data path designation. (Example: MMDDYY.S)

Click the "Check Sequence Icon" (Red Check mark and 3 auto-sampler vials picture). Make sure the box next to the statement "Overwrite Existing Data Files" is NOT checked. Click OK and view the sequence. Double check the vial numbers, data path designation and sequence file name. Printing this document is optional.

Again, click the "Save Sequence Icon" (3 ¼ inch Floppy Disk and 3 auto-sampler vials picture).

3. RUN SEQUENCE-DATA ACQUISITION

Click the "Run Sequence Icon" (Yellow stick figure in the running position and 3 auto-sampler vials picture). This will start the instrument injecting samples. Watch the first sample injection to ensure the auto-sampler is functioning properly.

4. DATA ANALYSIS

CLICK – Save Method (if different from method being sought)
CLICK – Load Method
CLICK - Load Data File
CLICK - Quantitate
CLICK – Calculate
CLICK – View
CLICK- QEDIT QUANT RESULTS
Go to DFTPP – CLICK
Review responses, ions, and RT
Go to Suspected Drug(s) – CLICK
Review responses, ions, and RT
Perform background subtraction if necessary

QDel any non-matching compounds

COMPARE Q SPECTRA WITH REFERENCE SPECTRA
CLICK – Spectrum
Perform background subtraction if necessary
CLICK – Display Reference Spectra
Review responses, ions, and RT of Sample spectrum (top) and Reference spectrum (bottom)

NOTE: The retention times for standards (reference spectra) and unknowns will be within +/- 0.25 minutes. Library matches for confirmations should be made to the HETLSDD Library, if possible, or any other approved available reference libraries, including SWGDRUG, Cayman, NIST, and Cerilliant. Communication regarding which libraries are approved for use will be sent and documented by the Quality Manager.

CLICK - Qedit
CLICK – Graphics Report to the Printer
Exit (close QEDIT QUANT RESULTS window)
SAVE changes to Quantitation Results? CLICK – YES

S/N Report
When a S/N ratio >3:1 is not clearly evident from the Quant QEdit report a S/N report using the target ion for the compound in question may be generated and included in the file.

PRINT FINAL REPORT
CLICK – Quantitate
CLICK – Generate Report
Under "Quant Report Options": Style-**Summary**; Destination-**Printer**
CLICK - OK

TO PRINT TOTAL SCAN AND SPECIFIC SPECTRA
CHOOSE Peak of interest
Left CLICK on the peak
(Double right CLICK on the Mass Spectrum for library search report)
Go to File Menu
Select Print
Select Print Trace and Spectrum
CLICK – OK

5. GC-MS PROGRAMS (list not inclusive)

WBAMINE2
STER
RAPIDRUG
LSD
RAPIDSUBOXONE
THC

Maintenance of GC/MS (only required during relevant periods when the instrument is in use)

Daily Maintenance

- Perform a Tune Evaluation

Weekly Maintenance

- Run QC Check solution
    - QC Check solution will consist of a set mixture of at least three standards and DFTPP internal standard. The same mixture will be run every week and after any additional instrument maintenance or repairs.
    - Data will be entered into a tracking spreadsheet and monitored to evaluate instrument performance

Monthly Maintenance

- Copy all case work and methods data from the previous month and transfer it to the appropriately labeled folder on the external hard drive
- Run the computer defragment software
- Restart Computer
- Change liner, septum, and o-ring
- Clean syringe
- Check diffusion pump fluid level
- Check foreline pump oil level
- Perform an Autotune
    - Evaluate Tune Parameters to ensure conformance (See GC-MS Parameter Cheat Sheet).
    - Perform following mass spec maintenance

As Needed Maintenance

- Air and Water Check
    - Perform if instrument is opened for any maintenance
- Clean source and Auto-tune
- Change gold seal
- Change gas tank
- Change gas traps and purifiers
- Replace/trim column
- Lubricate Seals
- Change filaments

Every 6 Months

- Check calibration vial
- Replace foreline pump oil

Every 12 Months - Performed during PM, when possible

- Replace traps and filters
- Replace diffusion pump fluid

GC-MS AUTOTUNE Parameters

Tune Parameter	Specific Parameter	Acceptance Range
Mass Assignment	69.00. 219.00, 502.00 amu	±0.10 amu
Peak Widths	0.55 amu	±0.10 amu
Isotopic Ratios	70/69	0.5-1.6%
Isotopic Ratios	220/219	3.2-5.4%
Isotopic Ratios	503/502	7.9-12.3%
Air to 69	28, 32 amu	<10%
Water to 69	18 amu	<20%

APPENDIX B

OPERATION OF THE FTIR

5. FT-IR MAINTENANCE (only required during relevant periods when the instrument is in use)

WEEKLY MAINTENANCE – Perform in the below order

LASER ALIGNMENT AND VERIFICATION

Laser alignment and verification shall be performed at least once per week. To perform the test, click on the Experiment Setup button on the far left of the toolbar. The click on the "Diagnostics" tab, click the "Align" button for the automatic alignment to begin. When the laser has finished aligning, click the "Laser Verification" button. Follow the guide to remove the accessory and click OK. Click the "Start Verification" button and click "OK" once the verification has finished. There is no printed documentation for this maintenance.

ATR Check

Load the Smart iTX accessory (the ATR accessory) and select "ValPro Qualification" from the "Analyze" menu. From the drop-down menu, select "Smart iTX accessory-PHEUR" and click the "Qualify" button on the far right. A pop-up will be displayed asking to collect background, click "OK" to collect background. Follow the on screen guide to load the polystyrene, lower/lock the arm in place and click "OK". Once the check is finished, the report will be displayed on the screen. Click "Print" and then "Close". Review the report for any issues and submit to the Quality Manager for review.

POLYSTYRENE CHECK

A scan of a traceable polystyrene film shall be done at least once per week. The polystyrene system check will be performed in the same manner as casework analysis, outlined in the previous section, and evaluated using the peaks in the table below. All five (5) peaks must fall within +/- 4 cm^{-1} for the instrument to pass. If any peak(s) fall outside the 'passing range' the instrument will be removed from service until all peaks fall within the defined passing range and the polystyrene must have a library match of 80 or greater to an approved library. Indicate if the check passed or failed on the print out and retain the printed spectra and library match in the maintenance binder.

Peaks for Comparison
694
1028
1491
1601
3025

MONTHLY MAINTENANCE

1. Run and print a new background

2. **Main System Check**
   Load the transmission accessory into the instrument**.** Select "ValPro Qualification" from the "Analyze" menu**.** From the drop-down menu, select "Nicolet iS20 KBr-Factory (CP, JP, PHEUR, PV, USP)-PV"**.** Click the "Qualify" button on the far right and allow the process to run for about 15 to 20 minutes. Once the check is finished, the report will be displayed on the screen. Click "Print" and then "Close". Review the report for any issues and submit to the Quality Manager for review.

3. **Software Checks**
   Select "ValPro Qualification" from the "Analyze" menu. From the drop-down menu, select "Quantification & Algorithms 2019-ALL". Click the "Qualify" button on the far right. Once the check is finished, the report will be displayed on the screen. Click "Print" and then "Close". Review the report for any issues and submit to the Quality Manager for review.

4. **Back up data**
   Copy all case work and maintenance data from the previous month and transfer it to the appropriately labeled folder on the external hard drive. Transfer that data to the K drive for network storage.

ANNUAL MAINTENANCE

Preventative Maintenance performed by competent vendor, when possible.

All maintenance shall be recorded on the maintenance checklist or the maintenance binder for the instrument. Copies of maintenance data from the current year are retained in a binder located near the FTIR. Maintenance data from past years are stored either in a designated folder in the Evidence Room or are archived or retained by the Quality Manager in digital format.

Revision Table		
Number	**Date**	**Changes Made**
1*	12/23/2020	Table of contents corrected Minor spelling, grammar, structural changes made throughout Section II C: updated to include extraction procedural blanks Section II E c and d: references to use of uncertainty associated with calibrated weights removed Section II Combined weight section: added the option of applying the most conservative number of weighing events to the total number of items tested Section III A 3: added language and examples of major and minor discrepencies Section III A 4: added guidance if seals are not intact Section III B 2: all hypergeometric sampling tables corrected, and references updated Section III C 1: Drugs.com added to tables Section IV A: items to be included in case notes updated Section IV B: chain of custody added to items included in casefile Section IV C: sample preparation updated to include a procedural blank and changed use of SDD solvent to "appropriate" solvent for blanks Section VI A: PPE updated, internal standard updated to include DFTPP as an option and methods updated to include Rapid Drug and Steroid as options Section VI B: step specifying preparing a tube as a procedural blank added Section VI B: PPE added Section VI C-G: title lettering updated for all, PPE section updated for all, additional solvent options added, extraction procedural blank language added, and sample preparation updated for all Section VI D: extraction tables updated to reflect current procedures Section VI E 2: weighing sample language removed Appendix A 4: language about subtraction added, reference to HETLSDD library added, S/N report section added Revision Table added
2	3/15/21	Section III, B, 2 was added to include a reserve weight definition and procedure

		Section VC was updated to note that sample solution guidance could be found in the Specific Analytical Procedures sections.
3	04/29/2021	References to Marijuana were removed throughout and replaced with either cannabis or delta-9 THC. Report wording was included in Section VI A to address reporting of cannabis samples.
4	03/08/2022	• Updated evidence room numbering throughout • Section II B updated to include the process for updating standards on the GC/MS and FTIR • Section III A updated to state initials shall be required across the seal of a package, and noted if not present. Updated to include evidence photos • Removed UoM procedure as there is a separate policy document. Section was revised to reference separate policy. • Section III C 1 was updated to include the limitations of a Category A technique. • Updated analysis of syringes throughout due to updated statute. Will be analyzed customer request only now. • Blank requirement added to section VI A 3 • Minor formatting, grammar, and spelling updates throughout • Section IV B: updated following observed PT • Added SDD Mix C as option throughout • Section VI D: added requirement of taking the weight of at least one pill/capsule/films • Section VI E: Corrected preparation of LSD/LAMPA standards • Section VI F: Corrected blank preparation • Section VI G: added option for edibles procedure • Removed GHB method from GC/MS section • Updated FTIR Appendix to better match process
5	3/10/22	• Corrected Section II.B New Lot Number of Existing Compounds to require one print out from both instruments for the library entry review. • Corrected numbering issues in FTIR Appendix
6	03/29/22	• Section I A: updated to include a description of the evidence on the contract, and included a process for repackaging evidence in new packaging after examination • Section II D: Processing blanks updated to include procedure relating to updating of a standard

		• Section III A 3: Noted that chemical abbreviations are listed in the Chemical Hygiene Plan and that a state controlled camera should be used for photographing evidence • III A 4: updated to include that all seals shall be checked for initials and noted if not present • III B 3: updated to include deviations shall be communicated to customer and included on the COA • III C 5: updated to include guidance for reporting inconclusive samples • V : updated to include customer communications relating to deviations • V F: updated to include second option for mushroom extraction
7	04/25/2022	• Updated Iodo TLC Spray to list 8% Platinic Chloride
8	06/28/2022	• Updated sections relating to FTIR for implementation of new Nicolet iS20 FTIR instrument
9	07/01/2022	• Updated FTIR section to include required scan region and corrected the word wavenumber to resolution in the blank and sample evaluation section.
10	08/09/2022	• Section I. A. changed room number to room description to prepare for move. Updated guidance for resealing packages. • Section II. B. added reference to SOP Manual and Quality Manual for standard handling and storage.
11	10/01/2022	• Removed Color Testing, removed all TLC sprays and systems, with the exception of the general drug screen system and Iodo spray, and removed Quechers ediles extraction. These are no longer active procedures. • Section I A updated to clarify that samples taken of evidence for testing are not tracked on the chain of custody.
12	03/31/23	• Added S/N criteria as an acceptance criteria for GCMS compound evaluation • Updated sample calculation of UoM to clarify what should be considered • Added procedure for daily/weekly balance checks • Corrected table of contents and section headers to be consistent for Section VI • Section VI. G. Waste was added. Includes specific waste instructions for all processes • Reference to use of ether removed and replaced with SDD Mix.

13	10/23/23	• Clarification, grammar, and spelling throughout. • Added a 10% threshold for agreement between the initial sealed package weight recorded upon submission and the package weight when the analyst begin the case. • TLC and GCMS sections clarified throughout • Blank requirements clarified throughout • Classification of a residue clarified
14	12/28/23	• TLC procedure retired and removed from SOP • Clarification of GCMS analysis processes made required for confirmation • Addition of use of extraction worksheet for mushroom samples • Added acid/base extraction guidance and procedure • Updated GCMS maintenance schedule and tune acceptance criteria added • Added instrument maintenance shall only be required during days/weeks/months where the instrument is in use.

*revisions prior to Dec 23, 2020 are listed in the Version History section of SharePoint